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COMPOSITION OF MATTER TAILORING: SYSTEM I

RELATED APPLICATION

This application is a divisional of U.S. Serial No. 10/123,228, filed April 12, 2002, which is a continuation-in-part of U.S. Serial No. 09/416,720, filed October

5   13, 1999, now U.S. Patent No. 6,572,792, issued June 3, 2003, and a continuation-in-part of International Application No. PCT/US00/28549, which designated the United States and was filed on October 13, 2000, published in English, which is a continuation of U.S. Serial No. 09/416,720, filed October 13, 1999. The entire teachings of the above applications are incorporated herein by

10   reference.

BACKGROUND OF THE INVENTION

All matter has structure. The structure of matter emanates from the electronic structure of the elements of the periodic table. It is the electronic structure of the elements and the new electronic structures that arise as a

15   consequence of their combination in molecules that define the electronic state and character of matter. It is also the electronic structure that creates the properties identified and associated with elements and the matter that results from their combination and arrangement (e.g., molecules and matter).

Certain combinations of elements give rise to states of matter with

20   particularly desirable properties. For instance, certain states of matter have long-range order, which refers to matter that has repeating aligned chemical, electronic, or structural units. Example of such states of matter include surfactant membranes, crystals such as smectic liquid crystals and liquid crystalline polymers, and magnetic materials.

One means of imparting unique properties to a material involves adding carbon to the material. Depending on the parent material and on the amount of carbon added, carbon may remain dissolved in a material or may precipitate out to form discrete carbon structures.

5   SUMMARY OF THE INVENTION

The present invention relates to a new composition of matter comprised of 'p', 'd', and/or 'f' atomic orbitals, and a process for making the composition of matter. This new composition of matter can be distinguished by a change in energy, electronic properties, physical properties, and the like. X-ray fluorescence

10   spectroscopy is a preferred method of detecting and distinguishing new compositions of matter. The change in properties can be controlled to be transient, fixed, or adjustable (temporarily permanent) and includes properties such as mechanical, electrical, chemical, thermal, engineering, and physical properties, as well structural character of the composition of matter (e.g., alignment, orientation,

15   order, anisotropy, and the like).

The present invention includes manufactured metals and alloys characterized by the x-ray fluorescence spectrometry plots and elemental abundance tables (obtained from x-ray fluorescence analysis) contained herein.

The present invention is additionally a method of processing a metal or an

20   alloy of metals, comprising the steps of:

    (A.)   adding the metal or alloy to a reactor in one or more steps and melting said metal or alloy;

    (B.)   adding a carbon source to the molten metal or alloy and dissolving carbon in said molten metal or alloy, followed by removing the

25   undissolved carbon source;

    (C.)   increasing the temperature of the molten metal or alloy;

    (D.)   varying the temperature of the molten metal or alloy between two temperatures over one or more cycles;

    (E.)   adding a flow of an inert gas through the molten metal or alloy;

(F.) varying the temperature of the molten metal or alloy between two temperatures over one or more cycles;

(G.) adding a carbon source to the molten metal or alloy and further dissolving carbon in said molten metal or alloy, followed by removing the undissolved carbon source;

(H.) varying the temperature of the molten metal or alloy between two temperatures over one or more cycles, wherein the molten metal or alloy has a greater degree of saturation with carbon than in Step (F.);

(I.) stopping the flow of the inert gas;

(J.) varying the temperature of the molten metal or alloy between two temperatures over one or more cycles, wherein the molten metal or alloy has a greater degree of saturation with carbon than in Step (H.) and wherein an inert gas is added as the temperature is lowered and an inert gas, chosen independently, is added as the temperature is raised;

(K.) varying the temperature of the molten metal or alloy between two temperatures over one or more cycles, wherein the molten metal or alloy has a greater degree of saturation with carbon than in Step (J.) and wherein an inert gas is added as the temperature is lowered and an inert gas, chosen independently, is added as the temperature is raised;

(L.) stopping the flow of the inert gases;

(M.) varying the temperature of the molten metal or alloy between two temperatures over one or more cycles, wherein the molten metal or alloy has an equal or greater degree of saturation with carbon than in Step (K.); and

(N.) cooling the molten metal or alloy to room temperature, thereby obtaining a solidified manufactured metal or alloy.

Steps (D.), (F.), (H.), (J.), (K.), and (L.) of the present method are commonly referred to as "cycling steps" below. For purposes of the present invention, carbon "dissolved" in a metal is defined as carbon that has been solubilized in a molten

metal, adsorbed by a metal, reacted with a metal, or has otherwise interacted with a metal such that carbon is desorbed or transferred from a solid carbon source into a molten metal.

Preferably, the present invention is a method of processing copper, comprised of the steps described above.

The present invention also includes a method of processing a metal or an alloy of metals, comprising the steps of:

(A.) adding the metal or alloy to a reactor in one or more steps and melting said metal or alloy;

(B.) adding a carbon source to the molten metal or alloy and dissolving carbon in said molten metal or alloy, followed by removing the undissolved carbon source;

(C.) varying the temperature of the molten metal or alloy between two temperatures over two or more cycles;

(D.) adding a carbon source to the molten metal or alloy and further dissolving carbon in said molten metal or alloy, followed by removing the undissolved carbon source;

(E.) varying the temperature of the molten metal or alloy between two temperatures over two or more cycles, wherein the molten metal or alloy has a greater degree of saturation with carbon than in Step (D.); and

(F.) cooling the molten metal or alloy to room temperature, thereby obtaining a solidified manufactured metal or alloy;

further characterized by adding a flow of inert gas, before, during, or after one or more of Steps (B.) through (E.).

In another embodiment, the present invention is a method of processing copper, or other metal or alloy comprising:

(1.) contacting molten copper or other metal or alloy with a carbon source;

(2.) an iterative cycling process, wherein relative saturation of copper or other metal or alloy with carbon remains the same or increases independently with each cycle; and

(3.) cooling the molten copper or other metal or alloy to room

5 temperature, thereby obtaining a solidified manufactured copper or other metal or alloy.

Advantages of the present invention include a method of processing metals into new compositions of matter and producing and characterizing compositions of matter with altered physical and/or electrical properties.

10 BRIEF DESCRIPTION OF THE DRAWINGS

Figures 1A and 1B show non-contact magnetic force microscopy images of natural copper and manufactured copper, respectively.

Figure 2A shows non-contact magnetic force microscopy of manufactured copper.

15 Figure 2B shows scanning tunneling microscopy images of manufactured copper.

Figures 3A, 3B, and 4A and 4B show x-ray emission spectrometry images of natural copper and manufactured copper.

Figure 5A shows a non-contact magnetic force microscopy image of

20 manufactured copper. Figure 5B shows a x-ray emission spectroscopy image of manufactured copper.

Figures 6A and 6B show a plot of an x-ray fluorescence spectrometry comparison of manufactured copper and natural copper.

Figure 7 shows a plot of an X-ray fluorescence spectrometry in relation to

25 the direction of the scan.

Figure 8 shows a plot of a change in capacitance and voltage decay for a manufactured metal.

Figure 9 shows a plot of a change in voltage gradients for a molten manufactured metal as the position of an electrode within the melt is changed.

Figure 10 shows a plot of the observed voltage of a manufactured copper-nickel alloy, as measured in a molten state.

Figure 11 shows a plot of the observed voltage of a manufactured metal, as measured in a molten state.

5 Figure 12 shows a plot of a positive voltage signature and positive capacitance decay of a manufactured metal, as measured in a molten state.

Figure 13 shows a plot of a voltage decay profile of a manufactured metal, as measured in a molten state.

Figure 14 shows a plot of a neutral decay in voltage and capacitance, as 10 measured in a molten state.

Figure 15 shows a plot of a positive voltage signature and a negative capacitance decay of a manufactured metal, as measured in a molten state.

Figure 16 shows a plot of the voltage over time of a manufactured metal under pressure.

15 Figures 17A, 17B, 17C, 18A, 18B and 18C show optical and scanning electron microscopy images of manufactured copper.

Figures 19A, 19B, 20A and 20B show optical microscopy images of manufactured nickel.

Figures 21A, 21B and 21C show images of atomic force microscopy and 20 scanning tunneling microscopy of manufactured copper from an axial analysis.

Figures 22A, 22B and 22C show images of atomic force microscopy and non-contact magnetic force microscopy of manufactured copper from a radial analysis.

Figures 23A, 23B and 23C show images of discrete induced magnetism of 25 non-magnetic copper.

Figure 24 shows a plot of electrical susceptance for manufactured compositions in comparison to the compositions in its natural state.

Figure 25 shows a plot of x-ray fluorescence spectrometry for manufactured copper, on both the axial and radial faces of a block cut from the ingot prepared in 30 Example 1, as compared to a plot of x-ray fluorescence spectrometry for natural copper.

Figure 26 shows a plot of x-ray fluorescence spectrometry for manufactured copper in the region of the K_α band of aluminum, on the bottom face of a block cut from the ingot prepared in Example 1, as compared to a plot of x-ray fluorescence spectrometry for natural aluminum.

5   Figures 27A and 27B show plots of x-ray fluorescence spectrometry for manufactured nickel, on both the axial and radial faces of a block cut from the ingot prepared in Example 2, as compared to plots of x-ray fluorescence spectrometry for natural nickel.

Figure 28A shows a plot of x-ray fluorescence spectrometry for

10   manufactured nickel in the region of the K_α band of aluminum, on the axial and radial faces of a block cut from the ingot prepared in Example 2, as compared to a plot of x-ray fluorescence spectrometry for natural aluminum.

Figure 28B shows a plot of x-ray fluorescence spectrometry for manufactured nickel in the region of the K_α band of zirconium, on the axial and

15   radial faces of a block cut from the ingot prepared in Example 2, as compared to a plot of x-ray fluorescence spectrometry for natural zirconium.

Figure 29 shows a plot of x-ray fluorescence spectrometry for manufactured nickel in the region of the K_α band of sulfur, on all six faces of a block cut from the ingot prepared in Example 2, as compared to a plot of x-ray fluorescence

20   spectrometry for natural sulfur.

Figure 30 shows a plot of x-ray fluorescence spectrometry for manufactured nickel in the region of the K_α band of chlorine (from potassium chloride), on the axial and radial faces of a block cut from the ingot prepared in Example 2, as compared to a plot of x-ray fluorescence spectrometry for natural chlorine (from

25   potassium chloride).

Figures 31A and 31B show plots of x-ray fluorescence spectrometry for manufactured cobalt, on both the axial and radial faces of a block cut from the ingot prepared in Example 3, as compared to plots of x-ray fluorescence spectrometry for natural cobalt.

30   Figures 32A shows a plot of x-ray fluorescence spectrometry for manufactured cobalt in the region of the K_α band of aluminum, on the axial and

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radial faces of a block cut from the ingot prepared in Example 3, as compared to a plot of x-ray fluorescence spectrometry for natural aluminum.

Figure 32B shows a plot of x-ray fluorescence spectrometry for manufactured cobalt in the region of the K_α band of iron, on the axial and radial faces of a block cut from the ingot prepared in Example 3, as compared to a plot of x-ray fluorescence spectrometry for natural iron.

Figure 33A shows a plot of x-ray fluorescence spectrometry for manufactured cobalt in the region of the K_α band of chlorine (from potassium chloride), on the axial and radial faces of a block cut from the ingot prepared in Example 3, as compared to a plot of x-ray fluorescence spectrometry for natural chlorine (from potassium chloride).

Figure 33B shows a plot of x-ray fluorescence spectrometry for manufactured cobalt in the region of the K_α band of zirconium, on the axial and radial faces of a block cut from the ingot prepared in Example 3, as compared to a plot of x-ray fluorescence spectrometry for natural zirconium.

Figure 34 shows a plot of x-ray fluorescence spectrometry for manufactured cobalt in the region of the K_α band of manganese, on the axial and radial faces of a block cut from the ingot prepared in Example 3, as compared to a plot of x-ray fluorescence spectrometry for natural manganese.

Figure 35 shows a plot of x-ray fluorescence spectrometry for a manufactured copper/silver/gold alloy in the region of the K_α band of copper, on the axial and radial faces of a block cut from the ingot prepared in Example 4, as compared to a plot of x-ray fluorescence spectrometry for natural copper.

Figure 36 shows a plot of x-ray fluorescence spectrometry for a manufactured copper/silver/gold alloy in the region of the K_α band of gold, on the axial and radial faces of a block cut from the ingot prepared in Example 4, as compared to a plot of x-ray fluorescence spectrometry for natural silver.

Figure 37 shows a plot of x-ray fluorescence spectrometry for a manufactured copper/silver/gold alloy in the region of the K_α band of silver, on the axial and radial faces of a block cut from the ingot prepared in Example 4, as compared to a plot of x-ray fluorescence spectrometry for natural gold.

Figure 38 shows a plot of x-ray fluorescence spectrometry for a manufactured tin/lead/zinc alloy in the region of the K_α band of tin, on the axial and radial faces of a block cut from the ingot prepared in Example 5, as compared to a plot of x-ray fluorescence spectrometry for natural tin.

5   Figure 39 shows a plot of x-ray fluorescence spectrometry for a manufactured tin/lead/zinc alloy in the region of the K_α band of zinc, on the axial and radial faces of a block cut from the ingot prepared in Example 5, as compared to a plot of x-ray fluorescence spectrometry for natural zinc.

Figure 40 shows a plot of x-ray fluorescence spectrometry for a manufactured tin/lead/zinc alloy in the region of the K_α band of lead, on the axial and radial faces of a block cut from the ingot prepared in Example 5, as compared to a plot of x-ray fluorescence spectrometry for natural lead.

Figure 41 shows a plot of x-ray fluorescence spectrometry for a manufactured tin/sodium/magnesium/potassium alloy in the region of the K_α band of potassium (from potassium chloride), on the axial and radial faces of a block cut from the ingot prepared in Example 6, as compared to a plot of x-ray fluorescence spectrometry for natural potassium (from potassium chloride).

Figure 42 shows a plot of x-ray fluorescence spectrometry for a manufactured tin/sodium/magnesium/potassium alloy in the region of the K_α band of tin, on the axial and radial faces of a block cut from the ingot prepared in Example 6, as compared to a plot of x-ray fluorescence spectrometry for natural tin.

Figure 43 shows a plot of x-ray fluorescence spectrometry for a manufactured tin/sodium/magnesium/potassium alloy in the region of the K_α band of magnesium (using magnesium oxide), on the axial and radial faces of a block cut from the ingot prepared in Example 6, as compared to a plot of x-ray fluorescence spectrometry for natural magnesium (using magnesium oxide).

Figure 44 shows a plot of x-ray fluorescence spectrometry for a manufactured tin/sodium/magnesium/potassium alloy in the region of the K_α band of sodium (using $AlNa_3F_6$), on the axial and radial faces of a block cut from the ingot prepared in Example 6, as compared to a plot of x-ray fluorescence spectrometry for natural sodium (using $AlNa_3F_6$).

Figures 45A and 45B show plots of x-ray fluorescence spectrometry for manufactured silicon, on both the axial and radial faces of a block cut from the ingot prepared in Example 7, as compared to plots of x-ray fluorescence spectrometry for natural silicon.

5   Figure 46A shows a plot of x-ray fluorescence spectrometry for manufactured silicon in the region of the K_α band of aluminum, on the axial and radial faces of a block cut from the ingot prepared in Example 7, as compared to a plot of x-ray fluorescence spectrometry for natural aluminum.

Figure 46B shows a plot of x-ray fluorescence spectrometry for 10   manufactured silicon in the region of the K_α band of titanium, on the axial and radial faces of a block cut from the ingot prepared in Example 7, as compared to a plot of x-ray fluorescence spectrometry for natural titanium.

Figure 47A shows a plot of x-ray fluorescence spectrometry for manufactured silicon in the region of the K_α band of sulfur, on the axial and radial 15   faces of a block cut from the ingot prepared in Example 7, as compared to a plot of x-ray fluorescence spectrometry for natural sulfur.

Figure 47B shows a plot of x-ray fluorescence spectrometry for manufactured silicon in the region of the K_α band of chlorine (from potassium chloride), on the axial and radial faces of a block cut from the ingot prepared in 20   Example 7, as compared to a plot of x-ray fluorescence spectrometry for natural chlorine (from potassium chloride).

Figure 48A shows a plot of x-ray fluorescence spectrometry for manufactured silicon in the region of the K_α band of gallium (from gallium oxide), on the axial and radial faces of a block cut from the ingot prepared in Example 7, as 25   compared to a plot of x-ray fluorescence spectrometry for natural gallium (from gallium oxide).

Figure 48B shows a plot of x-ray fluorescence spectrometry for manufactured silicon in the region of the K_α band of potassium (from potassium chloride), on the axial and radial faces of a block cut from the ingot prepared in 30   Example 7, as compared to a plot of x-ray fluorescence spectrometry for natural potassium (from potassium chloride).

Figures 49A and 49B show plots of x-ray fluorescence spectrometry for manufactured iron, on both the axial and radial faces of a block cut from the ingot prepared in Example 8, as compared to plots of x-ray fluorescence spectrometry for natural iron.

5   Figure 50A shows a plot of x-ray fluorescence spectrometry for manufactured iron in the region of the K_α band of aluminum, on the axial and radial faces of a block cut from the ingot prepared in Example 8, as compared to a plot of x-ray fluorescence spectrometry for natural aluminum.

Figure 50B shows a plot of x-ray fluorescence spectrometry for 10   manufactured iron in the region of the K_α band of zirconium, on the axial and radial faces of a block cut from the ingot prepared in Example 8, as compared to a plot of x-ray fluorescence spectrometry for natural zirconium.

Figure 51A shows a plot of x-ray fluorescence spectrometry for manufactured iron in the region of the K_α band of sulfur, on the axial and radial 15   faces of a block cut from the ingot prepared in Example 8, as compared to a plot of x-ray fluorescence spectrometry for natural sulfur.

Figure 51B shows a plot of x-ray fluorescence spectrometry for manufactured iron in the region of the K_α band of chlorine (from potassium chloride), on the axial and radial faces of a block cut from the ingot prepared in 20   Example 8, as compared to a plot of x-ray fluorescence spectrometry for natural chlorine (from potassium chloride).

Figure 52 shows a plot of x-ray fluorescence spectrometry for a manufactured iron/vanadium/chromium/manganese alloy in the region of the K_α band of chromium (using chromium(III) oxide), on the axial and radial faces of a 25   block cut from the ingot prepared in Example 9, as compared to a plot of x-ray fluorescence spectrometry for natural chromium (using chromium (III) oxide).

Figure 53 shows a plot of x-ray fluorescence spectrometry for a manufactured iron/vanadium/chromium/manganese alloy in the region of the K_α band of iron, on the axial and radial faces of a block cut from the ingot prepared in 30   Example 9, as compared to a plot of x-ray fluorescence spectrometry for natural iron.

Figure 54 shows a plot of x-ray fluorescence spectrometry for a manufactured iron/vanadium/chromium/manganese alloy in the region of the K_α band of vanadium, on the axial and radial faces of a block cut from the ingot prepared in Example 9, as compared to a plot of x-ray fluorescence spectrometry for

5 natural vanadium.

Figure 55 shows a plot of x-ray fluorescence spectrometry for a manufactured iron/vanadium/chromium/manganese alloy in the region of the K_α band of manganese, on the axial and radial faces of a block cut from the ingot prepared in Example 9, as compared to a plot of x-ray fluorescence spectrometry for

10 natural manganese.

Figure 56 shows a plot of x-ray fluorescence spectrometry for a manufactured iron/vanadium/chromium/manganese alloy in the region of the K_α band of sulfur, on all six sides of a block cut from the ingot prepared in Example 9.

Figure 57 shows a plot of x-ray fluorescence spectrometry for a

15 manufactured nickel/tantalum/hafnium/tungsten alloy in the region of the K_α band of tantalum, on the axial and radial faces of a block cut from the ingot prepared in Example 10, as compared to a plot of x-ray fluorescence spectrometry for natural tantalum.

Figure 58 shows a plot of x-ray fluorescence spectrometry for a

20 manufactured nickel/tantalum/hafnium/tungsten alloy in the region of the K_α band of tungsten, on the axial and radial faces of a block cut from the ingot prepared in Example 10, as compared to a plot of x-ray fluorescence spectrometry for natural tungsten.

Figure 59 shows a plot of x-ray fluorescence spectrometry for a

25 manufactured nickel/tantalum/hafnium/tungsten alloy in the region of the K_α band of hafnium, on the axial and radial faces of a block cut from the ingot prepared in Example 10, as compared to a plot of x-ray fluorescence spectrometry for natural hafnium.

Figure 60 shows a plot of x-ray fluorescence spectrometry for a

30 manufactured nickel/tantalum/hafnium/tungsten alloy in the region of the K_α band of sulfur, on all six faces of a block cut from the ingot prepared in Example 10.

Figure 61 shows a plot of x-ray fluorescence spectrometry for a manufactured nickel/tantalum/hafnium/tungsten alloy in the region of the K_α band of nickel, on the axial and radial faces of a block cut from the ingot prepared in Example 10, as compared to a plot of x-ray fluorescence spectrometry for natural nickel.

Figure 62 shows the general configuration of the ARL 8410 spectrometer.

DETAILED DESCRIPTION OF THE INVENTION

Electromagnetic chemistry is the science that affects the transfer and circulation of energy in many forms when induced by changes in electromagnetic energy. The theory of Electrodynamics of Moving Bodies (Einstein, 1905) mandates that when the electrodynamic components of the material are manipulated, changes in the energy levels within the atomic orbitals must be induced. These changes in the atomic orbitals are the vehicles by which changes in the (material) properties, such as the magnitude and/or the orientation, can occur. Alignment of the electrodynamic component induces effects that may result in significant changes in the underlying material species: (1) alignment of atoms with the resulting directionality of physical properties; (2) alignment of energy levels and the capability to produce harmonics, may establish physical properties conducive for energy transfer; (3) alignment of the electrodynamic component include the opening of pathways for free electron flow, and; (4) alignment of electrodynamic field phase orientation.

The present invention relates to new compositions of matter, referred to herein as "manufactured" metals or alloys of metals. A "manufactured" metal or alloy is a metal or alloy which exhibits a change in electronic structure, such as that seen in a fluid XRF spectrum. The American Heritage College Dictionary, Third Edition defines "fluid" as changing or tending to change.

Metals of the present invention are generally p, d, or f block metals. Metals include transition metals such as Group 3 metals (e.g., scandium, yttrium, lanthanum), Group 4 metals (e.g, titanium, zirconium, hafnium), Group 5 metals (vanadium, niobium, tantalum), Group 6 metals (e.g., chromium, molybdenum, tungsten), Group 7 metals (e.g., manganese, technetium, rhenium), Group 8 metals

(e.g., iron, ruthenium, osmium), Group 9 metals (e.g., cobalt, rhodium, iridium), Group 10 metals (nickel, palladium, platinum), Group 11 metals (e.g., copper, silver, gold), and Group 12 metals (e.g., zinc, cadmium, mercury). Metals of the present invention also include alkali metals (e.g., lithium, sodium, potassium,

5   rubidium, cesium) and alkaline earth metals (e.g., beryllium magnesium, calcium, strontium, barium). Additional metals include aluminum, gallium, indium, tin, lead, boron, germanium, arsenic, antimony, tellurium, bismuth, and silicon.

The present invention also includes alloys of metals. Alloys are typically mixtures of metals. Alloys of the present invention can be formed, for example, by

10   melting together two or more of the metals listed above. Preferred alloys include those comprised of copper, gold, and silver; tin, zinc, and lead; tin, sodium, magnesium, and potassium; iron, vanadium, chromium, and manganese; and nickel, tantalum, hafnium, and tungsten.

Carbon sources of the present invention include materials that are partially,

15   primarily, or totally comprised of carbon. Those carbon sources that are non-organic and comprised partially of carbon are primarily comprised of one or more metals. Suitable carbon sources include graphite rods, graphite powder, graphite flakes, fullerenes, diamonds, natural gas, methane, ethane, propane, butane, pentane, cast iron, iron comprising carbon, steel comprising carbon, and combinations

20   thereof. A preferred carbon source is a high purity (<5 ppm impurities) carbon source. Another preferred carbon source is a high purity (<5 ppm impurities) graphite rod. The carbon source is selected, in part, based on the system to which it is added. In one example, graphite rods and graphite flakes are added to copper, typically in a sequential manner. In another example, graphite rods and graphite

25   powder are added to iron, typically in a sequential manner.

Carbon sources can be contacted with molten metals for variable periods of time. The period of time the carbon source is in contact with molten metals is the time between adding the carbon source and removing the undissolved carbon source. The period of time can be from about 0.5 hours to about 12 hours, about 1

30   hour to about 10 hours, about 2 hours to about 8 hours, about 3 hours to about 6 hours, about 3.5 hours to about 4.5 hours, or about 3.9 hours to about 4.1 hours.

Alternatively, the period of time can be from about 5 minutes to about 300 minutes, about 10 minutes to about 200 minutes, about 20 minutes to about 120 minutes, about 30 minutes to about 90 minutes, about 40 minutes to about 80 minutes, about 50 minutes to about 70 minutes, or about 59 minutes to about 61 minutes.

5   A cycle of the present invention includes a period of time where the temperature and/or the degree to which a metal is saturated with carbon is varied. Over a period of time, varying the temperature involves a period of raising (or increasing) the temperature of a metal or alloy and a period when the temperature of a metal or alloy decreases (either passively, such as by heat transfer to the

10   surrounding environment, or actively, such as by mechanical means), in any order. Inert gas can be added during a cycle, except where it is specified that inert gas addition is ceased prior to that cycle. Increasing the temperature of the metal or alloy increases the amount of carbon that can be dissolved into that metal or alloy, which therefore decreases the degree to which the metal or alloy is saturated with

15   carbon (relative to the temperature and degree of carbon saturation when graphite saturation assemblies are removed the first time). Similarly, decreasing the temperature of the metal or alloy increases the (relative) degree to which the metal or alloy is saturated with carbon.

          The degree to which a metal is saturated with carbon varies over the course

20   of a method, as well as within each step. In Examples 1-14, the degree of carbon saturation varies between 70% and 95% in the first cycling step, between 70% and 95% in the second cycling step, between 101% and 103% in the third cycling step, between 104% and 107% in the fourth cycling step, between 108% and 118% in the fifth cycling step, and between 114% and 118% in the sixth cycling step. The

25   cycling steps correspond to Steps (D.), (F.), (H.), (J.), (K.), and (L.), respectively, of the method described in the third paragraph of the summary.

          One example of a method of the present invention can be described in terms of carbon saturation values. After a metal or alloy is added to a suitable reactor, establish the dissolved carbon level at 70% to 95% of the equilibrium saturation of

30   carbon for the thermodynamic state specified (e.g., T, P, composition) when the composition is in its natural state (hereinafter the equilibrium saturation of carbon is

referred to as "$[C]_{eqsat}$"). Identify temperature set points for 80% and 95% $[C]_{eqsat.}$ Vary the temperature between the predetermined set points, such that the temperature is decreased for 7 minutes and increased over 7 minutes per cycle, for 15 cycles. Next, establish a flow of argon. Vary the temperature between the

5   predetermined set points, such that the temperature is decreased for 7 minutes and increased over 7 minutes per cycle, for 5 cycles; the temperature should be maintained above 70% $[C]_{eqsat}$ at all times and maintained below 95% $[C]_{eqsat}$ at all times. The carbon level is raised to saturation (i.e., $[C]_{eqsat}$) with continued argon addition. Hold for 60 minutes at saturation (i.e., $[C]_{eqsat}$) with continued argon

10   addition. Raise the carbon level to $^+1\%_{wt}$ (i.e., $^+1\%_{wt}$ represents $1\%_{wt}$ above the saturation value as defined in its natural state) of $[C]_{eqsat}$ with continued argon addition and hold for 5 minutes. Vary the temperature for 20 cycles to remain between $^+1\%_{wt}$ and $^+3\%_{wt}$ of $[C]_{eqsat}$, such that the temperature is decreased over 9 minutes and increased over 9 minutes per cycle. Cease argon addition. Cool the

15   metal to $^+4\%_{wt}$ of $[C]_{eqsat}$. Vary the temperature for 4.5 cycles to remain between $^+4\%_{wt}$ and $^+7\%_{wt}$ of $[C]_{eqsat}$, such that the temperature is decreased over 3 minutes and increased over 5 minutes. Argon is added as the carbon saturation increases and nitrogen is added as carbon saturation decreases. Cool the metal to obtain $^+8\%_{wt}$ with continued argon addition. Vary the temperature over 15.5 cycles to remain

20   between $^+8\%_{wt}$ and $^+18\%_{wt}$ of $[C]_{eqsat}$, such that the temperature is decreased over 15 minutes and increased over 15 minutes. Argon is added as the carbon saturation increases and nitrogen is added as carbon saturation decreases. After the 15.5 cycles are complete, gas addition is halted. Perform one complete cycle by varying the temperature to remain between $^+18\%_{wt}$ to $^+14\%_{wt}$ of $[C]_{eqsat}$ (ending at $^+18\%_{wt}$), such

25   that the temperature is increased over 15 minutes and decreased over 15 minutes. Proceed immediately to a cool down that leads to solidification.

An iterative cycle process is a process comprising two or more cycles, whereby one or more of the cycles are carried out at a temperature below the carbon saturation point and one or more cycles are carried out at a temperature above the

30   carbon saturation point. For example, in Example 1, the first cycle is carried out between 2480°F and 2530°F, the second cycle is carried out between 2480°F and 2530°F, the third cycle is carried out at 2453°F and 2459°F, the fourth cycle is

carried out between 2441°F and 2450°F, the fifth cycle is carried out between 2406°F and 2438°F, and the sixth cycle is carried out between 2406°F and 2419°F. A cycle following an earlier cycle can have the identical temperature range as the earlier cycle, a partially overlapping temperature range with the earlier cycle, a

5   temperature range above that of the earlier cycle, or a temperature cycle below that of the earlier cycle. Partially overlapping temperature ranges includes ranges where one temperature range falls within the limits of a second temperature range (e.g., 2406°F to 2438°F and 2406°F to 2419°F). Preferably, during an iterative cycling process, the degree to which the metal or alloy is saturated with carbon increases

10   over the process.

Cycles of the present invention can vary in duration. The duration of a cycle can vary among cycles in a step. A cycle duration is, for example, about 2 minutes to about 90 minutes, about 3 minutes to about 67 minutes, about 5 minutes to about 45 minutes, about 8 minutes to about 30 minutes, about 10 minutes to about 20

15   minutes, about 14 minutes to about 18 minutes, about 7 minutes to about 9 minutes, about 13 minutes to about 15 minutes, about 17 minutes to about 19 minutes, about 28 minutes to about 32 minutes, or about 29 minutes to about 31 minutes.

A cycle can be symmetry or asymmetric. In a symmetric cycle, the period of increasing the metal or alloy temperature is equal to the period of decreasing the

20   metal or alloy temperature. In an asymmetric cycle, the period of increasing the metal or alloy temperature is different than the period of decreasing the metal or alloy temperature. For an asymmetric cycle, the period of increasing the metal or alloy temperature can be longer than or shorter than the period of decreasing the metal or alloy temperature.

25   For example, in a cycle lasting about 7 minutes to about 9 minutes, the temperature can be increased for about 3 minutes and the temperature can be decreased for about 5 minutes. If the cycle lasts about 13 minutes to about 15 minutes, the temperature can be increased for about 7 minutes and the temperature can be decreased for about 7 minutes. If the cycle lasts about 17 minutes to about

30   19 minutes, the temperature can be increased for about 9 minutes and the temperature can be decreased for about 9 minutes. If the cycle lasts about 29

minutes to about 31 minutes, the temperature can be increased for about 15 minutes and the temperature can decreased for about 15 minutes.

The number of cycles in a step is generally an integer or half-integer value. For example, the number of cycles in a step can be one or more, one to forty, or one to twenty. The number of cycles can be 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, 12, 13, 14, 15, 16, 17, 18, 19, 20, 21, 22, 23, 24, 25, 26, 27, 28, 29, 30, 31, 32, 33, 34, 35, 36, 37, 38, 39, or 40. Alternatively, the number of cycles in a step can be 0.5, 1.5, 2.5, 3.5, 4.5, 5.5, 6.5, 7.5, 8.5, 9.5, 10.5, 11.5, 12.5, 13.5, 14.5, 15.5, 16.5, 17.5, 18.5, 19.5, 20.5, 21.5, 22.5, 23.5, 24.5, 25.5, 26.5, 27.5, 28.5, 29.5, or 30.5. In a step comprising a half-integer or a non-integer quantity of cycles, either heating or cooling can occur first.

After the initial heating step, the temperature of a metal or an alloy is sufficiently high, such that the temperature is equal to or greater than the solidus temperature. The solidus temperature varies depending on the metal or the alloy, and the amount of carbon dissolved therein. The temperature at the end of Step (F.) of the third paragraph of the summary is typically about 900°F to about 3000°F, but varies from metal to metal. For example, the temperature at the end of Step (F.) can be about 1932°F to about 2032°F, about 1957°F to about 2007°F, or about 1932°F to about 2467°F for copper; about 2368°F to about 2468°F, about 2393°F to about 2443°F, or about 2368°F to about 2855°F for nickel; about 2358°F to about 2458°F or about 2373°F to about 2423°F, or about 2358°F to about 2805°F for cobalt; about 1932°F to about 2032°F, about 1957°F to about 2007°F, or about 1932°F to about 2467°F for a copper/gold/silver alloy; about 399°F to about 499°F, about 424°F to about 474°F, or about 399°F to about 932°F for a tin/lead/zinc alloy; about 399°F to about 499°F, about 424°F to about 474°F, or about 399°F to 932°F for a tin/sodium/potassium/magnesium alloy; about 2550°F to about 2650°F, about 2575°F to about 2625°F, or about 2550°F to about 2905°F for silicon; about 2058°F to about 2158°F, about 2073°F to about 2123°F, or about 2058°F to about 2855°F for iron; about 2058°F to about 2158°F, about 2073°F to about 2123°F, or about 2058°F to about 2855°F for an iron/vanadium/chromium/manganese alloy; or

2368°F to about 2468°F, about 2393°F to about 2443°F, or about 2368°F to about 2855°F for a nickel/tantalum/hafnium/ tungsten alloy.

Inert gas or gases can be added during a step. Inert gases are chosen, independently, from the group consisting of argon, nitrogen, helium, neon, xenon,

5 krypton, hydrogen, and mixtures thereof. When an inert gas is added during a step, the inert gas (or mixture thereof) can change from cycle-to-cycle or within a cycle

For purposes of the present invention, inert gases used for purging, particularly in the backspace of a reactor are generally considered separately from the other inert gases. Nitrogen is typically added continuously through a method of

10 the present invention, irrespective of whether "inert gas" flow into the metal is started or stopped. In one example, a nitrogen flow is maintained throughout an entire method, such that a nitrogen pressure of about 0.4-0.6 psi, or about 0.5 psi is maintained.

At the end of the instant methods, the molten metal or alloy is cooled. The

15 metal or alloy is cooled, at minimum, to a temperature below the solidus temperature. Preferably, the metal or alloy is cooled to room or ambient temperature. Such cooling can include gradual and/or rapid cooling steps. Gradual cooling typically includes cooling due to heat exchange with air or an inert gas over 1 to 72 hours, 2 to 50 hours, 3 to 30 hours, or 8 to 72 hours. Rapid cooling, also

20 known as quenching, typically includes an initial cooling with air or an inert gas to below the solidus temperature, thereby forming a solid mass, and placing the solid mass into a bath comprising a suitable fluid such as tap water, distilled water, deionized water, other forms of water, inert gases (as defined above), liquid nitrogen or other suitable liquified gases, a thermally-stable oil (e.g., silicone oil) or organic

25 coolant, and combinations thereof. The bath should contain a suitable quantity of liquid at a suitable temperature, such that the desired amount of cooling occurs.

Methods of the present invention are carried out in a suitable reactor. Suitable reactors are selected depending on the amount of metal or alloy to be processed, mode of heating, extent of heating (temperature) required, and the like.

30 A preferred reactor in the present method is an induction furnace reactor, which is capable of operating in a frequency range of 0 kHz to about 10,000 kHz, 0 kHz to

about 3,000 kHz, or 0 kHz to about 1,000 kHz. Reactors operating at lower frequencies are desirable for larger metal charges, such that a reactor operating at 0-3,000 kHz is generally suitable for 20 pound metal charges and a reactor operating at 0-1,000 kHz is generally suitable for 5000 pound metal charges.

5   Typically, reactors of the present method are lined with a suitable crucible. Crucibles are selected, in part, based on the amount of metal or alloy to be heated and the temperature of the method. Crucibles selected for the present method typically have a capacity from about five pounds to about five tons. One preferred crucible is comprised of 89.07% Al_2O_3, 10.37% SiO_2, 0.16% TiO_2, 0.15% Fe_2O_3,

10   0.03% CaO, 0.01% MgO, 0.02% Na_2O_3, and 0.02% K_2O, and has a 9" outside diameter, a 7.75" inside diameter, and a 14" depth. A second preferred crucible is comprised of 99.68% Al_2O_3, 0.07% SiO_2, 0.08% Fe_2O_3, 0.04% CaO, and 0.12% Na_2O_3, and has a 4.5" outside diameter, a 3.75" inside diameter and a 10" depth.

          After being subjected to a process of the present invention, metals and alloys

15   can be analyzed by a variety of techniques, including chemical and physical methods. A preferred analytical method is x-ray fluorescence spectrometry. X-ray fluorescence spectrometry is described in "X-Ray Fluorescence Spectrometry", by George J. Havrilla in "Handbook of Instrumental Techniques for Analytical Chemistry," Frank A. Settle, Ed., Prentice-Hall, Inc: 1997, which is incorporated

20   herein by reference.

          XRF spectrometry is a well-known and long-practiced method, which has been used to detect and quantify or semi-quantify the elemental composition (for elements with $Z \geq 11$) of solid and liquid samples. This technique benefits from minimal sample preparation, wide dynamic range, and being nondestructive.

25   Typically, XRF data are not dependent on which dimension (e.g., axial or radial) of a sample was analyzed. Accuracy of less than 1% error can generally be achieved with XRF spectrometry, and the technique can have detection limits of parts per million.

          XRF spectrometry first involves exciting an atom, such that an inner shell

30   electron is ejected (e.g., the photoelectric effect). Upon ejection of an electron, an outer shell electron will "drop" down into the lower-energy position of the ejected inner shell electron. When the outer shell electron "drops" into the lower-energy

inner shell, x-ray energy is released. Typically, an electron is ejected from the K, L, or M shell and is replaced by an electron from the L, M, or N shell. Because there are numerous combinations of ejections and replacements possible for any given element, x-rays of several energies are emitted during a typical XRF experiment.

5   Therefore, each element in the Periodic Table has a standard pattern of x-ray emissions after being excited by a sufficiently energetic source, since each such element has its own characteristic electronic state. By matching a pattern of emitted x-ray energies to values found in tables, such as those on pages 10-233 to 10-271 of "Handbook of Chemistry and Physics, 73rd Edition," edited by D. R. Lide, CRC

10   Press, 1992, which is incorporated herein by reference, one can identify which elements are present in a sample. In addition, the intensity of the emitted x-rays allows one to quantify the amount of an element in a sample.

There are two standard variations of the XRF technique. First, as an energy-dispersive method (EDXRF), the XRF technique uses a detector such as a Si(Li)

15   detector, which is capable of simultaneously measuring the energy and intensity of x-ray photons from an array of elements. EDXRF is well-suited for rapid acquisition of data to determine gross elemental composition. Typically, the detection limits for EDXRF are in the range of tens to hundreds of parts-per-million. A wavelength-dispersive technique (WDXRF) is generally better-suited for analyses

20   requiring high accuracy and precision. WDXRF uses a crystal to disperse emitted x-rays, based on Bragg's Law. Natural crystals, such as lithium fluoride and germanium, are commonly used for high-energy (short wavelength) x-rays, while synthetic crystals are commonly used for low-energy (longer wavelength) x-rays. Crystals are chosen, in part, to achieve desired resolution, so that x-rays of different

25   energies are disperse to distinguishable 2θ angles. WDXRF can either measure x-rays sequentially, such that a WDXRF instrument will step through a range of 2θ angles in recording a spectrum, or there will be detectors positioned at multiple 2θ angles, allowing for more rapid analysis of a sample. Detectors for WDXRF commonly include gas ionization and scintillation detectors. A further description

30   of the use WDXRF technique in the present invention can be found in Example 1. Results from EDXRF and results from WDXRF can be compared by determining the relationship between a 2θ angle and the wavelength of the corresponding x-ray

(e.g., using Bragg's Law) and converting the wavelength into an energy (e.g., energy equals wavelength multiplied by Planck's constant).

Analysis of emitted x-rays can be carried out automatically or semi-automatically, such as by using a software package (e.g., UniQuant, which is sold by Omega Data Systems BV, Veldhoven, The Netherlands) for either EDXRF or WDXRF. UniQuant is used for standardless semi-quantitative to quantitative XRF analysis using the intensities measured by a sequential x-ray spectrometer. The software package unifies all types of samples into one analytical program. The UniQuant software program is highly effective for analyzing samples for which no standards are available. Sample preparation is usually minimal or not required at all. Samples can be of very different natures, sizes and shapes. Elements from fluorine or sodium up to uranium, or their oxide compounds, can be analyzed in samples such as a piece of glass, a screw, metal drillings, lubricating oil, loose fly ash powder, polymers, phosphoric acid, thin layers on a substrate, soil, paint, the year rings of trees, and, in general, those samples for which no standards are available. The reporting is in weight % along with an estimated error for each element.

In software packages such as UniQuant, an XRF spectrum is composed of data channels. Each data channel corresponds to an energy range and contains information about the number of x-rays emitted at that energy. The data channels can be combined into one coherent plot to show the number or intensity of emitted x-rays versus energy or 2θ angle (the 2θ angle is related to the wavelength of an x-ray), such that the plot will show a series of peaks. An analysis of the peaks by one skilled in the art or the software package can identify the correspondence between the experimentally-determined peaks and the previously-determined peaks of individual elements. For an element, peak location (i.e., the centroid of the peak with respect to energy or 2θ angle), peak profile/ shape, peak creation, and peak fluidity would be expected to be essentially the same, within experimental error, for any sample containing the element. If the same quantity of an element is present in two samples, intensity will also be essentially the same, excepting experimental error and matrix effects.

A typical software package is programmed to correlate certain data channels with the emitted x-rays of elements. Quantification of the intensity of emitted x-rays is accomplished by integrating the XRF spectrum over a number of data channels. Based on the measured intensities and the previously-compiled data on

5   elements, the software package will integrate over all data channels, correlate the emitted x-ray intensities, and will then calculate the relative abundance or quantity of elements which appear to be present in a sample, based upon comparison to the standards. Ideally, the relative abundances will total 100% prior to normalization. However, for a variety of reasons, such as improper or insufficient calibration, the

10   relative abundances will not total 100% prior to normalization. Another reason that the relative abundances of elements do not total 100% prior to normalization is that a portion of the XRF spectrum falls outside of the data channels that the software package correlates with an element (i.e., a portion of the XRF spectrum is not recognized as belonging to an element and is not included in the relative abundance

15   calculation). In this case, the relative abundances will likely total less than 100% prior to normalization. Representative examples of relative abundance data are presented in Table 4, which includes the results of XRF analyses of the products of Examples 1-10, as processed by a Uniquant software package.

X-ray emission spectrometry (XES), a technique analogous to XRF, also

20   provides electronic information about elements. In XES, a lower-energy source is used to eject electrons from a sample, such that only the surface (to several micrometers) of the sample is analyzed. Similar to XRF, a series of peaks is generated, which corresponds to outer shell electrons replacing ejected inner shell electrons. The peak shape, peak fluidity, peak creation, peak intensity, peak

25   centroid, and peak profile are expected to be essentially the same, within experimental error and matrix effects, for two samples having the same composition.

A new composition of matter of the present invention can manifest itself as a transient, adjustable, or permanent change in energy and/or associated properties,

30   as broadly defined. Property change can be exhibited as or comprise a change in: (1) structural atomic character (e.g., XES/XRF peak creation, peak fluidity, peak intensity, peak centroid, peak profile or shape as a function of material/sample

orientation, atomic energy level(s), and TEM, STM, MFM scans); (2) electronic
character (e.g., electron electromagnetic interactions, electromagnetic field
position/orientation, energy gradients, Hall effect, voltage, capacitance, voltage
decay rate, voltage gradient, voltage signature including slope of decay and/or
5   change of slope decay, voltage magnitude, voltage orientation); (3) structural
molecular or atomic character (e.g, SEM, TEM, STM, AFM, LFM, and MFM scans,
optical microscopy images, and structural orientation, ordering, long range
alignment/ordering, anisotropy); (4) physical constants (e.g., color, crystalline form,
specific rotation, emissivity, melting point, boiling point, density, refractive index,
10   solubility, hardness, surface tension, dielectric, magnetic susceptibility, coefficient
of friction, x-ray wavelengths); (5) physical properties (e.g., mechanical, chemical,
electrical, thermal, engineering, and the like); and, (6) other changes that
differentiate naturally occurring materials from manufactured materials created by
inducing a change in matter.

15   1. <u>Structural Atomic Character</u>

In the sections below, certain analyses have been conducted where a block
of a manufactured product (e.g., a metal or an alloy) has been cut from a larger
piece. In these analyses, the axial direction or an axial trace refers to a side of the
block that was originally parallel to the side walls of a reactor. The radial direction
20   or a radial trace refers to a side of the block that was originally parallel with the top
or bottom of a reactor. A metal block can also contain micro- or macro-voids that
can be analyzed.

Figure 1A shows a non-contact, magnetic force microscopy image of natural
copper, the control standard, and Figure 1B shows a new composition of matter:
25   manufactured copper, which is identified by an altered and aligned electromagnetic
network. Figure 2A shows a non-contact, magnetic force microscopy (MFM) scan
and Figure 2B shows a scanning tunneling microscopy (STM) scan. Individually,
and from differing vantage points, these scans show the outline of the changed
electromagnetic energy network. The MFM scan shows the radial trace while the
30   STM scan shows the axial trace.

XES analysis of the control standard compared to the atomically altered (i.e., manufactured) state are shown in Figures 3A, 3B, 4A, and 4B. Manufactured copper in the axial direction exhibits similar composition to natural copper (i.e., $99.98\%_{wt}$), but radial scans exhibit new peaks in the region close to naturally

5   occurring S, Cl, and K. The shifting centroid of the observed peaks from the natural species (i.e., S, Cl, K) confirm electronic change in the atomic state of the base element; as does the non-contact MFM void scan (compare Figure 1B with Figure 5A). Conventional chemical analysis performed using a LECO (IR) analyzer confirmed the absence of sulfur at XES lower detection limits. LECO analysis

10   confirmed sulfur concentration at 7.8 ppm; this analysis was consistent with the manufacture's batch product analysis of 7.0 ppm S.

Figure 5B compares the XES radial scan of manufactured copper to that of a void space within the same material. An underlying change in atomic character can be inferred from a dramatic change in signal count/intensity and a non-contact MFM

15   of the void space (Figure 5A). MFM evidence highlights the structure and its changed orientation and alignment compared to the control MFM (Figure 1A).

High precision XRF imaging shows that manufactured copper has a K_α line in the vicinity of 110.7 degrees (the 2θ angle). Since 110.7 degrees is the location of natural sulfur's K_α line, this K_α line is referred to herein as a "sulfur-like" K_α line.

20   This is the K_α line that would be expected if detectable quantities of sulfur were present; however, an IR LECO analysis of this sample showed that there was no sulfur was present (<10 ppm) in the sample. The presence of this line indicates an electronic structure change, which has shifted the two theta degree position of the K_α line compared to natural copper (Figures 6A and 6B). Several other figures

25   indicate the presence of unexpected K_α lines for elements not present in the sample (e.g., Figure 27 shows the presence of a significant aluminum-like K_α line for a sample containing 99.98% copper). Figure 7 shows an increase in signal intensity dependent upon which side of a homogeneous block of sample was analyzed, as well as a shifting K_α centroid. These data demonstrate microscopically the bulk

30   anisotropy later identified in the manufactured sample as does the MFM scans (Figures 1B and 5A).

2. Electronic Character

Manipulation of the electrodynamic components affecting the orientation of a manufactured metal's or alloy's electromagnetic field can enable the observance of a Hall voltage (V_H). Manipulation of the electrodynamic components enables

5   intensification of electromagnetic field affording charge concentration on the surface of the atoms within the bulk as opposed to the bulk surface of the bath. Properties that reflect field repositioning can include changing capacitance and voltage decay rate (Figure 8) and voltage gradients (Figure 9) within a conducting bulk media.

10   Figure 10 shows the V_H observed in a copper-nickel alloy. Voltage decay exhibited two distinct decay rates, indicative of two controlling mechanisms. A positive voltage signature with a positive capacitance decay (i.e., capacitance accumulation) is shown in Figure 11.

Control and manipulation of the charge signature (e.g., V_H profile,

15   capacitance slope, voltage slope) provides evidence of the alteration, and manipulation of the underlying electronic state. Figure 12 shows a positive voltage signature and a positive capacitance decay. Additionally, the voltage decay profile has changed: one profile has a negative slope while the other has a neutral slope. Further change in the electronic structure enables the slope of the second voltage

20   decay profile to become positive (Figure 13); note also the change in slope of capacitance decay. The metal system shown in Figure 14 has an electronic structure change that result in a nearly neutral decay in voltage and capacitance. Measurements were repeated four times. Figure 15 shows that the voltage can actually become negative, indicating that the orientation can also be manipulated.

25   Figure 16 shows the phenomena can be observed under pressure. Table 1 is an XRF analysis using a Uniquant software package that shows a multiplicity of energetically contiguous X-ray atomic energy levels. One energetically contiguous series is represented by Sm, Eu, Gd, Tb; the other is represented by P, S, So (i.e., sulfur as an oxide), Cl, and Ar. Table 2 is an XRF analysis using a Uniquant

30   software package that shows an energetically contiguous series as Al, Si, P, S, So (i.e., sulfur as an oxide), Cl, Ar, K, and Ca. Table 3 represents an experiment that utilized the same starting material as Tables 1 and 2, however, the reported amount

or abundance of copper after processing differs from the other tables. The differing relative abundances of elements observed in Tables 1-3 are believed to correspond to the unexpected peaks seen in many of the XRF plots.

3. Structural Molecular/Atomic Character

5   New compositions of matter can be electronically modified to induce long range ordering/alignment. In one new composition of matter, long range ordering was induced in oxygen-free high conductivity (OFHC) copper. Optical microscopy and SEM imaging of the material verifies the degree and extent of long range ordering achieved (Figures 17A, 17B, 17C, 18A, 18B, and 18C). Under similar

10   electronic conditions, long range ordering was induced in high purity (99.99 %$_{wt}$) nickel. Figures 19A, 19B, 20A, and 20B show the optical microscopy imaging of the manufactured nickel material. A comparison of alignment is shown in-run, at two different points during processing, which highlights the adjustability of the altered electronic state of the manufactured nickel.

15   Extensive atomic force microscopy and non-contact MFM imaging of electronically altered OFHC copper shows views of structural configurations from a different perspective (Figure 21A, 21B, 21C, 22A, 22B, and 22C). Non-contact MFM imaging shows clear pattern repetition and intensity of the manufactured copper when compared to the natural copper. The manufactured copper represents a

20   new composition of matter derived from natural copper, and the manufactured copper exhibits anisotropic behavior.

4. Physical Constants

     In one sequence of new compositions of matter, color changes in OFHC copper were induced. The variation in color over four (4) new matter compositions

25   ranged from black (two intensities) to copper (2 intensities) to gold (one intensity) to silver (one intensity). While not being bound by theory, the alteration of copper's electronic state along the continuum enables the new composition of matter's color to be adjusted along the continuum.

     In another sequence of new compositions of matter, changes in the hardness

30   of OFHC copper were induced. The variation in diamond pyramid hardness

between different manufactured copper samples ranged from about 25 to 90 (or 3 to 9 times higher than natural copper). Hardness change was anisotropic.

In another new composition of matter, magnetism was induced in a high purity, non-magnetic metal copper (e.g., 99.98 $\%_{wt}$) in its elemental form (Figures
5   23A and 23B).

5. Physical Properties

In one sequence of new compositions of matter, ductility changes were induced in a high purity, ductile copper (99.98%$_{wt}$) in its elemental form. The variation in the engineering physical property of ductility ranged from brittle to
10   semi-ductile to ductile to extremely ductile over four (4) new matter compositions.

In one new composition of matter, the electrical reactance was increased approximately 3% above that of natural copper over the frequency range of 0 Hz to 100 kHz. In another new composition of matter, electrical susceptance was increased approximately 20% above 99.98%$_{wt}$ copper of the same chemical
15   composition (i.e., the copper in its natural state). In another new composition of matter, electrical susceptance was decreased approximately 25% below 99.98%$_{wt}$ copper of the same chemical composition (i.e., the copper in its natural state). Electrical susceptance for these new matter compositions compared to the control standard (the material in its natural state) is shown in Figure 24.

20   6. Additional Differentiations

In one sequence of new compositions of matter, which all used the same raw materials, consumables, utilities, and materials of construction, the sum of element concentrations identified by XRF analysis varied considerably. Variations in elemental abundance determined by XRF Uniquant prior to normalization over three
25   (3) new matter compositions were 99.5 $\%_{wt}$ (Table 3), 96.0 $\%_{wt}$ (Table 2), and 90.6 $\%_{wt}$ (Table 1). The apparent loss of matter between the recognized elemental structures and the manufactured elemental structures differentiates naturally occurring materials from materials with modified electronic structures (i.e., a new composition of matter).

While not being bound by theory, Applicant believes the conservation of energy requires that all mass, independent of magnitude and/or configuration, character, and/or dimension can be characterized by the allowed set of mathematical poles (defined as the operation zurn) and further characterized by the set of

5   mathematical poles coalesced (defined as the isozurn value). An adjustment or manipulation of the zurn invokes a change in the isozurn value to a value different than its naturally occurring value, and accounts for the contribution of its rest state value, thereby modifying the electronic structure that defines the natural state. A change in the isozurn value to a value different than that which specifies the natural

10   state denotes a change in the underlying electronic state of the specified species.

A change in the isozurn value is typically noted at the subatomic, atomic, or molecular level. While not being bound by theory, the complexity of energy interactions is believed to often impede single variable isolation. In these cases, a change in the electronic state of the specified species typically manifests itself as a

15   change in a property value(s) from the naturally occurring state (e.g., the unaltered entropic driven ground state). Typical changes denoting a change in property value, which depart from the property value specifying its naturally occurring state, dictate a change in the isozurn value of that state.

Definitions of Acronyms

20   AO – Atomic Orbital

SEM – Scanning electron microscopy

TEM – Tunneling Electron Microscopy

STM – Scanning Tunneling Microscopy

AFM – Atomic Force Microscopy

25   LFM – Lateral Force Microscopy

MFM – Magnetic Force Microscopy

XES – X-ray Emission Spectrometry

XRF – X-ray Fluorescence Spectrometry

EXEMPLIFICATION

EXAMPLE 1

EXPERIMENTAL PROCEDURE FOR COPPER RUN 14-01-01

A cylindrical alumina-based crucible (99.68% Al_2O_3, 0.07% SiO_2, 0.08%
5   Fe_2O_3, 0.04% CaO, 0.12% Na_2O_3; 4.5" O.D. x 3.75" I.D. x 10" depth) of a 100
pound induction furnace reactor (Inductotherm) fitted with a 75-30R Powertrak
power supply was charged with 2500 g copper (99.98% purity) through its charging
port. The reactor was fitted with a graphite cap and a ceramic liner (i.e., the
crucible, from Engineering Ceramics). During the entire procedure, a slight positive
10   pressure of nitrogen (~0.5 psi) was maintained in the reactor using a continuous
backspace purge. The reactor was heated to the metal charge liquidus point plus
300°F, at a rate no greater than 300°F/hour, as limited by the integrity of the
crucible. The induction furnace operated in the frequency range of 0 kHz to 3000
kHz, with frequency determined by a temperature-controlled feedback loop
15   implementing an Omega Model CN300 temperature controller. Upon reaching
2300°F, the reactor was charged with an additional 2143 g copper over an hour.
The temperature was again increased to 2462°F again using a rate no greater
than 300°F/hour. When this temperature was reached, graphite saturation
assemblies (3/8" OD, 36" long high purity (<5 ppm impurities) graphite rods) were
20   inserted to the bottom of the copper charge through ports located in the top plate.
The copper was held at 2462°F for 4 hours. Every 30 minutes during the hold
period, an attempt was made to lower the graphite saturation assemblies as
dissolution occurred. As the copper became saturated with carbon, the graphite
saturation assemblies were consumed. After the 4 hour hold period was complete,
25   the graphite saturation assemblies were removed.
The reactor temperature was increased to 2480°F over 7 minutes. The
temperature was then varied between 2480°F and 2530°F for 15 cycles. Each cycle
consisted of raising the temperature continuously over 7 minutes and lowering the
temperature continuously over 7 minutes. After the 15 cycles were completed, a gas
30   addition lance was lowered into the molten metal to a position approximately 2"

from the bottom of the reactor and a 0.15 L/min flow of argon was begun. The temperature of the copper was varied over another 5 cycles between 2480°F and 2530°F.

     After the fifth cycle, the reactor temperature was lowered to 2462°F over a 10 minute period with continued argon addition. The graphite saturation assemblies were reinstalled in the copper and remained there for 1 hour. The graphite saturation assemblies were removed.

     The reactor temperature was lowered to 2459°F over 5 minutes. The reactor was held at this temperature for 5 minutes with continued argon addition. The temperature was then varied between 2459°F and 2453°F over 20 cycles. Each cycle consisted of lowering the temperature continuously over 9 minutes and raising the temperature continuously over 9 minutes. The argon addition ceased after completion of the 20 cycles.

     The reactor temperature was lowered to 2450°F over 5 minutes. The temperature was varied between 2450°F and 2441°F over 4 ½ cycles. Each cycle consisted of lowering the temperature continuously over 5 minutes and raising the temperature continuously over 3 minutes. In addition, while raising the temperature, a 0.15 L/min flow of argon was added, and while lowering the temperature, a 0.15 L/min flow of nitrogen was added.

     The reactor temperature was lowered to 2438°F over 5 minutes. The temperature was varied between 2438°F and 2406°F for 15.5 cycles. Each cycle consisted of lowering the temperature continuously over 15 minutes and raising the temperature continuously over 15 minutes. In addition, while raising the temperature, a 0.15 L/min flow of argon was added, and while lowering the temperature, a 0.15 L/min flow of nitrogen was added. All gas addition, except for the purge of nitrogen ceased after the 15.5 cycles were completed.

     The temperature was varied between 2406°F and 2419°F for one cycle. The cycle consisted of raising the temperature continuously over 15 minutes and lowering the temperature continuously over 15 minutes. The gas addition lance was removed .

The reactor temperature was cooled by lowering the induction furnace power to 1 kW or less as the ingot cooled. The copper was then cooled to approximately ambient temperature in water.

ANALYTICAL PROTOCOLS

5   X-ray Fluorescence

An ARL 8410 XRF was used to analyze each of the sample ingots. An ARL 8410 is a sequential wavelength dispersive spectrometer (WDS). Specific emission lines are used to determine the presence or absence, and the concentrations of various elements. Each characteristic x-ray line is measured in sequence by the

10   instrument by controlling the instrument geometry.

Figure 62 shows the general configuration of the ARL 8410 spectrometer. The WDS spectrometer relies on the fundamentals of x-ray diffraction, when x-ray fluorescence occurs when matter is bombarded by a stream of high-energy incident x-ray photons. When the incident X-radiation strikes the sample, the incident x-rays

15   may be absorbed, scattered, or transmitted for the measurement of the fluorescent yield.

The ARL 8410 utilizes an end-window rhodium (Rh) x-ray tube. The end-window is composed of Be, and holds the tube at high vacuum. The filaments are heated giving off electrons by thermoionic emission. This beam of electrons then

20   bombards the target Rh anode across a 10-70 keV voltage potential. Thus, primary x-rays are produced during the collision. The emitted x-ray spectrum consists of (1) "Continuum" or "Bremstrahlung" radiation, (2) Characteristic x-ray lines of the target material (e.g., K and L series), and (3) Characteristic lines from any contaminants. Thus, the primary spectrum appears as a series of sharp intense peaks

25   arrayed over a broad hump of continuum radiation. The ARL is equipped with and uses two types of photon detectors, the Flow Proportional Counter (FPC) and the Scintillation Counter (SC).

The manufactured metal samples are prepared by cutting a cube shape (approximately 1.1875") from the center of the cooled ingot. An axial edge and a

30   radial edge are then denoted. To provide a smooth surface for analysis, the axial and radial faces are sequentially polished. The sample faces are sanded to 400 grit,

then a polishing wheel is employed with 600 grit paper. Finally, a ≤ 1 μm polishing compound completes the smoothing process. The sample is then cleaned with iso-propyl alcohol and placed in a sample cassette/holder. The sample holder is then loaded into the XRF.

5   The orientation of the detector crystal with respect to the sample and the photon detector is controlled synchronously such that characteristic x-ray lines can be accurately measured. A sequential measurement consists of positioning the diffraction crystal at a given theta (diffraction angle) and the detector at two-theta and counting for a given period of time. The crystal and detector are then rotated to

10   a different angle for the next characteristic x-ray line.

Uniquant Version 2 software, developed by Omega Data Systems is used to control the crystal and detector placement and provides the data reduction algorithms for each analytical protocol. The sample results include an elemental composition list along with the associated concentrations for each sample.

15   Measurement of Grain Sizes

When the grain sizes exceeded the size discernible with the human eye, the grain size (average span distance) was measured using a micrometer. When the grain sizes were not discernible via the naked eye, standard acid etching was performed and then optical microscopy was utilized to measure and characterize the

20   grain.

Measurement of Magnetism

The magnetic properties of the manufactured ingots were tested via three methods.

<u>Magnetic Attraction:</u> An 1/8" diameter neodymium iron boron magnet was scanned

25   consistently and uniformly across the surface of the ingot to detect areas of attraction. Areas of attraction were then noted at specific sites on the surface.

<u>Attraction to Iron:</u> The attraction of iron filings to specific points on the ingot were quantified by enumerating the number of filings retained on the ingot surface in a vertical or upside-down orientation.

Gauss Measurement: The magnetic behavior of various points on the ingot were quantified via the use of a F. W. Bell 4048 Gauss meter.

Measurement of Chemical Reactivity

The manufactured ingots were subjected to various chlorine ligands, including NaCl, NaOCl, HCl, and chlorinated organics to semi-quantitatively access their reactivity to ligated chlorine. The formation of reaction products was recorded, then reaction products were removed from the reaction site, weighed and elemental composition verified via XRF.

ANALYTICAL RESULTS

An x-ray fluorescence analysis of the copper sample is provided in Figure 26, with the K_α peak of a copper control standard shown for reference.

An x-ray fluorescence analysis of the copper sample is provided in Figure 27, with the K_α peak of an aluminum control standard shown for reference.

Summary data showing the apparent elemental composition of the product of Example 1 is shown in Tables 5-13, as was measured by an XRF analysis using a Uniquant software package. The apparent elemental composition of the product varies by position, which is indicated in each table.

The manufactured copper exhibited large grain sizes and different coloration on each grain, which caused the surface to appear iridescent. The axial (top) face of the ingot appeared glassy, while the sides were metallic in appearance (due to anisotropic behavior). The color on both the axial and radial surfaces mimicked that of natural copper (i.e., not the intense reds or dark browns observed in other manufactured coppers, for example, Examples 11-14). On the axial surface, unique demarcations were observed. The ingot had some internal void areas, which were open to the top surface. No unexpected magnetic activity or chemical reactivity were recorded.

EXAMPLE 2

EXPERIMENTAL PROCEDURE FOR NICKEL RUN 14-01-04

A cylindrical alumina-based crucible (99.68% Al_2O_3, 0.07% SiO_2, 0.08% Fe_2O_3, 0.04% CaO, 0.12% Na_2O_3; 4.5" O.D. x 3.75" I.D. x 10" depth) of a 100

5   pound induction furnace reactor supplied by Inductotherm, was fitted with a 75-30R Powertrak power supply and charged with 2500 g nickel (99.97% purity) and 100 g of graphite carbon through its charging port. The reactor was fitted with a graphite cap with a ceramic liner (i.e. the crucible, from Engineering Ceramics). During the entire procedure, a slight positive pressure of nitrogen (~0.5 psi) was maintained in

10   the reactor using a continuous backspace purge. The reactor was heated to the metal charge liquidus point, over a rate no greater than 300°F/hour, as limited by the integrity of the crucible. The induction furnace operated in a frequency range of 0 kHz to 3000 kHz, with frequency determined by a temperature-controlled feedback loop implementing an Omega Model CA 300 temperature controller. Upon

15   reaching 2800°F, the reactor was charged with an additional 2700 g nickel over an hour.

The temperature was again increased to 2850°F again using a rate no greater than 300°F/hour. When this temperature was reached, graphite saturation assemblies (3/8" OD, 36" long high purity [<5 ppm impurities] graphite rods) were

20   inserted to the bottom of the nickel charge through ports located in the top plate. The nickel was held at 2850°F for 4 hours. Every 30 minutes during the hold period, an attempt was made to lower the graphite saturation assemblies as dissolution occurred. As the nickel became saturated with carbon, the graphite saturation assemblies were consumed. After the 4 hour hold period was complete,

25   the graphite saturation assemblies were removed.

The reactor temperature was increased to 3256°F over 7 minutes. The temperature was then varied between 2950°F and 3256°F for 15 cycles. Each cycle consisted of lowering the temperature continuously over 7 minutes and raising the temperature continuously over 7 minutes. After the 15 cycles were completed, a gas

30   addition lance was lowered and a 0.15 L/min flow of argon was begun. The

temperature of the nickel was varied over another 5 cycles between 2950°F and 3256°F.

After the fifth cycle, the reactor temperature was lowered to 2850°F over a 10 minute period with continued argon addition. The graphite saturation assemblies were reinstalled in the nickel and remained there for 1 hour. The graphite saturation assemblies were removed.

The reactor temperature was lowered to 2829°F over 5 minutes. The reactor was held at this temperature for 5 minutes with continued argon addition. The temperature was then varied between 2790°F and 2829°F over 20 cycles. Each cycle consisted of lowering the temperature continuously over 9 minutes and raising the temperature continuously over 9 minutes. The argon addition ceased after completion of the 20 cycles.

The reactor temperature was lowered to 2770°F over 5 minutes. The temperature was varied between 2710°F and 2770°F over 4 ½ cycles. Each cycle consisted of lowering the temperature continuously over 5 minutes and raising the temperature continuously over 3 minutes. In addition, while raising the temperature, a 0.15 L/min flow of argon was added, and while lowering the temperature, a 0.15 L/min flow of nitrogen was added.

The reactor temperature was lowered to 2691°F over 5 minutes. The temperature was varied between 2492°F and 2691°F for 15.5 cycles. Each cycle consisted of lowering the temperature continuously over 15 minutes and raising the temperature continuously over 15 minutes. In addition, while raising the temperature, a 0.15 L/min flow of argon was added, and while lowering the temperature, a 0.15 L/min flow of nitrogen was added. All gas addition, except for the purge of nitrogen ceased after the 15.5 cycles were completed.

The temperature was varied between 2571°F and 2492°F for one cycle. The cycle consisted of raising the temperature continuously over 15 minutes and lowering the temperature over 15 minutes. The gas addition lance was removed.

The reactor temperature was slowly cooled by lowering the induction furnace power to 1 KW or less as the ingot cooled. The nickel was then cooled to approximately ambient temperature in water.

ANALYTICAL PROTOCOLS

XRF, grain size, magnetism, and chemical reactivity measurements were carried out by the procedures described in Example 1.

ANALYTICAL RESULTS

5   An x-ray fluorescence analysis of the nickel sample is provided in Figures 28A and 28B, with the K_α and L_α peaks of a nickel control standard shown for reference.

An x-ray fluorescence analysis of the nickel sample is provided in Figure 29A, with the K_α peak of an aluminum control standard shown for reference.

10   An x-ray fluorescence analysis of the nickel sample is provided in Figure 29B, with the K_α peak of a zirconium control standard shown for reference.

An x-ray fluorescence analysis of the nickel sample is provided in Figure 30A, with the K_α peak of a sulfur control standard shown for reference.

An x-ray fluorescence analysis of the copper sample is provided in Figure
15   30B, with the K_α peak of an chlorine (from potassium chloride) shown for reference.

Summary data showing the apparent elemental composition of the product of Example 2 is shown in Tables 14-16, as was measured by an XRF analysis using a Uniquant software package. The apparent elemental composition of the product varies by position, which is indicated in each table.

20   The manufactured nickel retained a large amount of refractory on its exterior surface after retrieval from the reactor. The retained refractory was attributed to either surface attraction or reaction with the high content of Al_2O_3 in the refractory. The ingot did not crack with handling, but did have an internal void. The visible radial surface appeared duller in than the axial (top) face, again demonstrating
25   anisotropic physical properties. The ingot demonstrated no unexpected chemical reactivity after removal from the reaction system.

EXAMPLE 3

EXPERIMENTAL PROCEDURE FOR COBALT RUN 14-01-05

A cylindrical alumina-based crucible (99.68% Al_2O_3, 0.07% SiO_2, 0.08% Fe_2O_3, 0.04% CaO, 0.12% Na_2O_3; 4.5" O.D. x 3.75" I.D. x 10" depth) of a 100
5   pound induction furnace reactor supplied by Inductotherm and fitted with a 75-30R Powertrak power supply, was charged with 2176 g cobalt (99.8% purity) through its charging port. The reactor was fitted with a graphite cap with a ceramic liner from Engineering Ceramics. During the entire procedure, a slight positive pressure of nitrogen (~0.5 psi) was maintained in the reactor using a continuous backspace
10   purge. The reactor was heated to 2800°F over a minimum of 14 hours while the induction furnace operated in a frequency range of 0 kHz to 3000 kHz. Upon reaching 2700°F, the reactor was charged with an additional 3000 g cobalt over an hour.

When 2800°F was reached, graphite saturation assemblies were inserted to
15   the bottom of the cobalt charge through ports located in the top plate. The cobalt was held at 2800°F for 4 hours. Every 30 minutes during the hold period, an attempt was made to lower the graphite saturation assemblies as dissolution progressed. As the cobalt became saturated with carbon, the graphite saturation assemblies were consumed. After the 4 hour hold period was complete, the graphite
20   saturation assemblies were removed.

The reactor temperature was increased to 3086°F over 7 minutes. The temperature was then varied between 2875°F and 3086°F for 15 cycles. Each cycle consisted of lowering the temperature continuously over 7 minutes and raising the temperature continuously over 7 minutes. After the 15 cycles were completed, a gas
25   addition lance was lowered and a 0.15 L/min flow of argon was begun. The temperature of the cobalt was varied over another 5 cycles between 2875°F and 3086°F.

After the fifth cycle, the reactor temperature was lowered to 2800°F over a 10 minute period with continued argon addition. The graphite saturation assemblies
30   were reinstalled in the cobalt and remained there for 1 hour. The graphite saturation assemblies were removed.

The reactor temperature was lowered to 2785°F over 5 minutes. The reactor was held at this temperature for 5 minutes with continued argon addition. The temperature was then varied between 2689°F and 2785°F over 20 cycles. Each cycle consisted of lowering the temperature continuously over 9 minutes and raising

5   the temperature continuously over 9 minutes. The argon addition ceased after completion of the 20 cycles.

The reactor temperature was lowered to 2737°F over 5 minutes. The temperature was varied between 2689°F and 2737°F over 4 ½ cycles. Each cycle consisted of lowering the temperature continuously over 5 minutes and raising the

10 temperature continuously over 3 minutes. In addition, while raising the temperature, a 0.15 L/min flow of argon was added, and while lowering the temperature, a 0.15 L/min flow of nitrogen was added.

The reactor temperature was lowered to 2672°F over 5 minutes. The temperature was varied between 2498°F and 2672°F for 15.5 cycles. Each cycle

15 consisted of lowering the temperature continuously over 15 minutes and raising the temperature continuously over 15 minutes. In addition, while raising the temperature, a 0.15 L/min flow of argon was added, and while lowering the temperature, a 0.15 L/min flow of nitrogen was added. All gas addition, except for the purge of nitrogen ceased after the 15.5 cycles were completed.

20 The temperature was varied between 2570°F and 2498°F for one cycle. The cycle consisted of raising the temperature continuously over 15 minutes and lowering the temperature continuously over 15 minutes. The gas addition lance was removed.

The reactor temperature was cooled by lowering the induction furnace

25 power to 1 kW or less as the ingot cooled. The cobalt was then cooled to approximately ambient temperature in water.

ANALYTICAL PROTOCOLS

XRF, grain size, magnetism, and chemical reactivity measurements were carried out by the procedures described in Example 1.

ANALYTICAL RESULTS

An x-ray fluorescence analysis of the cobalt sample is provided in Figures 31A and 31B, with the K_α and L_α peaks of a cobalt control standard shown for reference.

5   An x-ray fluorescence analysis of the cobalt sample is provided in Figure 32A, with the K_α peak of an aluminum control standard shown for reference.

An x-ray fluorescence analysis of the cobalt sample is provided in Figure 32B, with the K_α peak of an iron control standard shown for reference.

An x-ray fluorescence analysis of the cobalt sample is provided in Figure 10   33A, with the K_α peak of a chlorine (from potassium chloride) control standard shown for reference.

An x-ray fluorescence analysis of the cobalt sample is provided in Figure 33B, with the K_α peak of a zirconium control standard shown for reference.

An x-ray fluorescence analysis of the cobalt sample is provided in Figure 15   34, with the K_α peak of a manganese control standard shown for reference.

Summary data showing the apparent elemental composition of the product of Example 3 is shown in Table 17, as was measured by an XRF analysis using a Uniquant software package.

The top (axial) face of the manufactured cobalt ingot exhibited many of the 20   recursive patterns observed in other manufactured ingots. The surface peaks are inconsistent with what would be expected given the forces of gravity during cooling. In addition, the shiny top face of the ingot exhibited an unexpected coloration, such that some of the faces had a distinct pink tint. While the top of the ingot was shiny, silver metallic, the sides of the ingot were matte silver in 25   appearance.

The manufactured cobalt ingot retained a small amount of refractory around its base. The ingot did not crack upon retrieval from the reaction system. No unexpected magnetic behavior or chemical reactivity were observed.

EXAMPLE 4

EXPERIMENTAL PROCEDURE FOR COPPER/GOLD/SILVER RUN 14-01-06

A cylindrical alumina-based crucible (99.68% Al_2O_3, 0.07% SiO_2, 0.08%
Fe_2O_3, 0.04% CaO, 0.12% Na_2O_3; 4.5" O.D. x 3.75" I.D. x 10" depth) of a 100
5   pound induction furnace reactor supplied by Inductotherm and fitted with a 75-30R
Powertrak power supply was charged with 2518 g copper (99.98% purity), plus
62.28 g each of gold (99.9999% pure) and silver (99.9999% pure) through its
charging port. The reactor was fitted with a graphite cap with a ceramic liner by
Engineering Ceramics. During the entire procedure, a slight positive pressure of
10   nitrogen (~0.5 psi) was maintained in the reactor using a continuous backspace
purge. The reactor was heated to 2300°F over a minimum of 12 hours while the
induction furnace operated in a frequency range of 0 kHz to 3000 kHz. Upon
reaching 2300°F, the reactor was charged with an additional 2000 g copper over an
hour.

15   The temperature was again increased to 2462°F again using a rate no greater
than 300°F/hour. When this temperature was reached, graphite saturation
assemblies were inserted to the bottom of the metal charge through ports located in
the top plate. The alloy was held at 2462°F for 4 hours. Every 30 minutes during
the hold period, an attempt was made to lower the graphite saturation assemblies.
20   As the alloy became saturated with carbon, the graphite saturation assemblies were
consumed. After the 4 hour hold period was complete, the graphite saturation
assemblies were removed.

The reactor temperature was increased to 2480°F over 7 minutes. The
temperature was then varied between 2480°F and 2530°F for 15 cycles. Each cycle
25   consisted of raising the temperature continuously over 7 minutes and lowering the
temperature continuously over 7 minutes. After the 15 cycles were completed, a gas
addition lance was lowered and a 0.15 L/min flow of argon was begun. The
temperature of the alloy was varied over another 5 cycles between 2480°F and
2530°F.

30   After the fifth cycle, the reactor temperature was lowered to 2462°F over a
10 minute period with continued argon addition. The graphite saturation assemblies

were reinstalled in the alloy and remained there for 1 hour. The graphite saturation assemblies were removed.

The reactor temperature was lowered to 2459°F over 5 minutes. The reactor was held at this temperature for 5 minutes with continued argon addition. The temperature was then varied between 2453°F and 2459°F over 20 cycles. Each cycle consisted of lowering the temperature continuously over 9 minutes and raising the temperature continuously over 9 minutes. The argon addition ceased after completion of the 20 cycles.

The reactor temperature was lowered to 2450°F over 5 minutes. The temperature was varied between 2441°F and 2450°F over 4 ½ cycles. Each cycle consisted of lowering the temperature continuously over 5 minutes and raising the temperature continuously over 3 minutes. In addition, while raising the temperature, a 0.15 L/min flow of argon was added, and while lowering the temperature, a 0.15 L/min flow of nitrogen was added.

The reactor temperature was lowered to 2438°F over 5 minutes. The temperature was varied between 2406°F and 2438°F for 15.5 cycles. Each cycle consisted of lowering the temperature continuously over 15 minutes and raising the temperature continuously over 15 minutes. In addition, while raising the temperature, a 0.15 L/min flow of argon was added, and while lowering the temperature, a 0.15 L/min flow of nitrogen was added. All gas addition, except for the purge of nitrogen ceased after the 15.5 cycles were completed.

The temperature was varied between 2406°F and 2419°F for one cycle. The cycle consisted of raising the temperature continuously over 15 minutes and lowering the temperature continuously over 15 minutes. The gas addition lance was removed.

The reactor temperature was cooled by lowering the induction furnace power to 1 kW or less as the ingot cooled. The copper/silver/gold was then cooled to approximately ambient temperature in water.

ANALYTICAL PROTOCOLS

XRF, grain size, magnetism, and chemical reactivity measurements were carried out by the procedures described in Example 1.

ANALYTICAL RESULTS

An x-ray fluorescence analysis of the copper/gold/silver alloy sample is provided in Figure 35, with the K_α peak of a copper control standard shown for reference.

5   An x-ray fluorescence analysis of the copper/gold/silver alloy sample is provided in Figure 36, with the K_α peak of a gold control standard shown for reference.

An x-ray fluorescence analysis of the copper/gold/silver alloy sample is provided in Figure 37, with the K_α peak of a silver control standard shown for

10   reference.

Summary data showing the apparent elemental composition of the product of Example 4 is shown in Tables 18-19, as was measured by an XRF analysis using a Uniquant software package. The apparent elemental composition of the product varies by position, which is indicated in each table.

15   The manufactured copper-based alloy exhibited uniform coloration in both the axial and radial directions. Prior to being quenched in water, the ingot exhibited significant iridescence on all surfaces. After being quenched, the intensity of iridescence diminished.

An unexpected feature of the ingot was the axial face crystal orientation. No

20   magnetic behavior was observed. The ingot did not crack or retain any refractory after retrieval from the reactor.

EXAMPLE 5

EXPERIMENTAL PROCEDURE FOR TIN/LEAD/ZINC 14-01-07

A cylindrical alumina-based crucible (99.68% Al_2O_3, 0.07% SiO_2, 0.08%

25   Fe_2O_3, 0.04% CaO, 0.12% Na_2O_3; 4.5" O.D. x 3.75" I.D. x 10" depth) of a 100 pound induction furnace reactor supplied by Inductotherm and fitted with a 75-30R Powertrak power supply was charged with 2562 g copper (99.9% purity), plus 854 g each of Lead (99+% pure) and Zinc (99.8% pure) through its charging port. The reactor was fitted with a graphite cap with a ceramic liner by Engineering Ceramics.

30   During the entire procedure, a slight positive pressure of nitrogen (~0.5 psi) was

maintained in the reactor using a continuous backspace purge. The reactor was heated to 932°F over a minimum of 4 hours at a rate no greater than 300°F/hour. The induction furnace operated in the frequency range of 0 kHz to 3000 kHz with frequency determined by a temperature-controlled feedback loop implementing an

5   Omega Model CN300 temperature controller.

When 932°F was reached, graphite saturation assemblies were inserted to the bottom of the metal charge through ports located in the top plate. The tin/lead/zinc alloy was held at 932°F for 4 hours. Every 30 minutes during the hold period, an attempt was made to lower the graphite saturation assemblies. As the

10   tin/lead/zinc alloy became saturated with carbon, the graphite saturation assemblies were consumed. After the 4 hour hold period was complete, the graphite saturation assemblies were removed.

The reactor temperature was increased to 968°F over 7 minutes. The temperature was then varied between 942°F and 968°F for 15 cycles. Each cycle

15   consisted of lowering the temperature continuously over 7 minutes and raising the temperature continuously over 7 minutes. After the 15 cycles were completed, a gas addition lance was lowered into the molten metal to a position approximately 2" from the bottom of the reactor and a 0.15 L/min flow of argon was begun. The temperature of the tin/lead/zinc alloy was varied over another 5 cycles between

20   942°F and 968°F.

After the fifth cycle, the reactor temperature was lowered to 932°F over a 10 minute period with continued argon addition. The graphite saturation assemblies were reinstalled in the tin/lead/zinc alloy and remained there for 1 hour. The graphite saturation assemblies were removed.

25   The reactor temperature was lowered to 930°F over 5 minutes. The reactor was held at this temperature for 5 minutes with continued argon addition. The temperature was then varied between 918°F and 930°F over 20 cycles. Each cycle consisted of lowering the temperature continuously over 9 minutes and raising the temperature continuously over 9 minutes. The argon addition ceased after

30   completion of the 20 cycles.

The reactor temperature was lowered to 924°F over 5 minutes. The temperature was varied between 918°F and 924°F over 4 ½ cycles. Each cycle

consisted of lowering the temperature continuously over 5 minutes and raising the temperature continuously over 3 minutes. In addition, while raising the temperature, a 0.15 L/min flow of argon was added, and while lowering the temperature, a 0.15 L/min flow of nitrogen was added.

5   The reactor temperature was lowered to 916°F over 5 minutes. The temperature was varied between 894°F and 916°F for 15.5 cycles. Each cycle consisted of lowering the temperature continuously over 15 minutes and raising the temperature over 15 minutes. In addition, while raising the temperature, a 0.15 L/min flow of argon was added, and while lowering the temperature, a 0.15 L/min

10   flow of nitrogen was added. All gas addition, except for the purge of nitrogen ceased after the 15.5 cycles were completed.

The temperature was varied between 894°F and 903°F for one cycle. The cycle consisted of raising the temperature continuously over 15 minutes and lowering the temperature continuously over 15 minutes. The gas addition lance was

15   removed.

The reactor temperature was cooled by lowering the induction furnace power to 1 kW or less as the ingot cooled. The tin/lead/zinc alloy was then cooled to approximately ambient temperature in water.

ANALYTICAL PROTOCOLS

20   XRF, grain size, magnetism, and chemical reactivity measurements were carried out by the procedures described in Example 1.

ANALYTICAL RESULTS

An x-ray fluorescence analysis of the tin/lead/zinc alloy sample is provided in Figure 38, with the K_α peak of a tin control standard shown for reference.

25   An x-ray fluorescence analysis of the tin/lead/zinc alloy sample is provided in Figure 39, with the K_α peak of a zinc control standard shown for reference.

An x-ray fluorescence analysis of the tin/lead/zinc alloy sample is provided in Figure 40, with the K_α peak of a lead control standard shown for reference.

Summary data showing the apparent elemental composition of the product

30   of Example 5 is shown in Tables 20-21, as was measured by an XRF analysis using

a Uniquant software package. The apparent elemental composition of the product varies by position, which is indicated in each table.

The manufactured tin-based alloy exhibited some stratification along the sides of the ingot. The top (axial) face and the side (radial) face did not appear

5 significantly different in coloration or appearance, and each had a matte finish. Like other manufactured alloy ingot, apparent peaks were exhibited on the axial face of the ingot.

The ingot did not have an internal void. No unexpected chemical activity or magnetic activity were recorded. The ingot did not crack upon retrieval from the

10 reactor and retained a small amount of refractory.

EXAMPLE 6

EXPERIMENTAL PROCEDURE FOR TIN/SODIUM, MAGNESIUM AND POTASSIUM 14-01-08

A cylindrical alumina-based crucible (99.68% Al_2O_3, 0.07% SiO_2, 0.08%

15 Fe_2O_3, 0.04% CaO, 0.12% Na_2O_3; 4.5" O.D. x 3.75" I.D. x 10" depth) of a 100 pound induction furnace reactor supplied by Inductotherm and fitted with a 75-30R Powertrak power supply, was charged with 2000 g tin (99.9% purity), plus 50 g each of sodium (99.8% pure), potassium (98% pure) and magnesium (99.98% pure) through its charging port. The reactor was fitted with a graphite cap with a ceramic

20 liner (i.e. the crucible, from Engineering Ceramics). During the entire procedure, a slight positive pressure of nitrogen (~0.5 psi) was maintained in the reactor using a continuous backspace purge. The reactor was heated to the metal charge liquidus point plus 300°F, at a rate no greater than 300° F/hour, as limited by the integrity of the crucible. The induction furnace operated in the frequency range of 0 kHz to

25 3000 kHz, with frequency determined by a temperature-controlled feedback loop implementing an Omega Model CN300 temperature controller. Upon reaching 900°F, the reactor was charged with an additional 2120 g Sn over an hour.

The temperature was again increased to 932°F again using a rate no greater than 300°F/hour. When this temperature was reached, graphite saturation

30 assemblies (3/8" OD, 36" long high purity (<5 ppm impurities) graphite rods) were

inserted to the bottom of the Sn/Na/K/Mg charge through ports located in the top plate. The Sn/Na/K/Mg alloy was held at 932°F for 4 hours. Every 30 minutes during the hold period, an attempt was made to lower the graphite saturation assemblies as dissolution occurred. As the Sn/Na/K/Mg alloy became saturated

5   with carbon, the graphite saturation assemblies were consumed. After the 4 hour hold period was complete, the graphite saturation assemblies were removed.

The reactor temperature was increased to 968°F over 7 minutes. The temperature was then varied between 942°F and 968°F for 15 cycles. Each cycle consisted of lowering the temperature continuously over 7 minutes and raising the

10   temperature continuously over 7 minutes. After the 15 cycles were completed, a gas addition lance was lowered into the molten metal to a position approximately 2" from the bottom of the reactor and a 0.15 L/min flow of argon was begun. The temperature of the Sn/Na/K/Mg alloy was varied over another 5 cycles between 942°F and 968°F.

15   After the fifth cycle, the reactor temperature was lowered to 932°F over a 10 minute period with continued argon addition. The graphite saturation assemblies were reinstalled in the tin/sodium/potassium/magnesium alloy and remained there for 1 hour. The graphite saturation assemblies were removed.

The reactor temperature was lowered to 930°F over 5 minutes. The reactor

20   was held at this temperature for 5 minutes with continued argon addition. The temperature was then varied between 918°F and 930°F over 20 cycles. Each cycle consisted of lowering the temperature continuously over 9 minutes and raising the temperature continuously over 9 minutes. The argon addition ceased after completion of the 20 cycles.

25   The reactor temperature was lowered to 924°F over 5 minutes. The temperature was varied between 918°F and 924°F over 4 ½ cycles. Each cycle consisted of lowering the temperature continuously over 5 minutes and raising the temperature continuously over 3 minutes. In addition, while raising the temperature, a 0.15 L/min flow of argon was added, and while lowering the

30   temperature, a 0.15 L/min flow of nitrogen was added.

The reactor temperature was lowered to 916°F over 5 minutes. The temperature was varied between 894°F and 916°F for 15.5 cycles. Each cycle

consisted of lowering the temperature continuously over 15 minutes and raising the temperature continuously over 15 minutes. In addition, while raising the temperature, a 0.15 L/min flow of argon was added, and while lowering the temperature, a 0.15 L/min flow of nitrogen was added. All gas addition, except for
5   the purge of nitrogen ceased after the 15.5 cycles were completed.

The temperature was varied between 894°F and 903°F for one cycle. The cycle consisted of raising the temperature continuously over 15 minutes and lowering the temperature continuously over 15 minutes. The gas addition lance was removed.

10   The reactor temperature was cooled by lowering the induction furnace power to 1 kW or less as the ingot cooled. The tin/sodium/magnesium/potassium alloy solidified into an ingot. After solidification, the alloy was cooled to approximately ambient temperature in water.

ANALYTICAL PROTOCOLS

15   XRF, grain size, magnetism, and chemical reactivity measurements were carried out by the procedures described in Example 1.

ANALYTICAL RESULTS

An x-ray fluorescence analysis of the tin/sodium/potassium/magnesium alloy sample is provided in Figure 41, with the K_α peak of a potassium (from
20   potassium chloride) control standard shown for reference.

An x-ray fluorescence analysis of the tin/sodium/potassium/magnesium alloy sample is provided in Figure 42, with the K_α peak of a tin control standard shown for reference.

An x-ray fluorescence analysis of the tin/sodium/potassium/magnesium alloy sample is provided in Figure 43, with the K_α peak of a magnesium control
25   standard shown for reference.

An x-ray fluorescence analysis of the tin/sodium/potassium/magnesium alloy sample is provided in Figure 44, with the K_α peak of a sodium (from $AlNa_3F_6$) control standard shown for reference.

Summary data showing the apparent elemental composition of the product of Example 6 is shown in Tables 22-23, as was measured by an XRF analysis using a Uniquant software package. The apparent elemental composition of the product varies by position, which is indicated in each table.

5   The ingot had a uniform dull matte finish on axial and radial surfaces (i.e., isotropic coloration). Minimal refractory was retained upon retrieval from the reactor. No internal voids were found in the ingot. No unexpected magnetic or chemical activity were observed.

EXAMPLE 7

10   EXPERIMENTAL PROCEDURE FOR SILICON 15-01-01

A cylindrical alumina-based crucible (99.68% Al_2O_3, 0.07% SiO_2, 0.08% Fe_2O_3, 0.04% CaO, 0.12% Na_2O_3; 4.5" O.D. x 3.75" I.D. x 10" depth) of a 100 pound induction furnace reactor (Inductotherm) fitted with a 75-30R Powertrak power supply was charged with 700 g Silicon (100.00% purity), through its

15   charging port. The reactor was fitted with a graphite cap and a ceramic liner (i.e., the crucible, from Engineering Ceramics). During the entire procedure, a slight positive pressure of nitrogen (~0.5 psi) was maintained in the reactor using a continuous backspace purge. The reactor was heated to the metal charge liquidus point plus 300°F, at a rate no greater than 300°F/hour as limited by the integrity of

20   the crucible. The induction furnace operated in the frequency range of 0 kHZ to 3000kHz, with frequency determined by a temperature-controlled feedback loop implementing an Omega Model CN 300 temperature controller. Upon reaching 2800°F, the reactor was charged with an additional 400 g Silicon again using a rate no greater than 300°F/hour.

25   The temperature was again increased to 2900°F again using a rate no greater than 300°F/hour. When this temperature was reached, graphite saturation assemblies (3/8" OD, 36" long high purity (5 ppm impurities) graphite rods) were inserted to the bottom of the Silicon charge through ports located in the top plate. The Silicon was held at 2900°F for 4 hours. Every 30 minutes during the hold

30   period, an attempt was made to lower the graphite saturation assemblies as

dissolution occurred. As the Silicon became saturated with carbon, the graphite saturation assemblies were consumed. After the 4 hour hold period was complete, the graphite saturation assemblies were removed.

The reactor temperature was increased to 2976°F over 7 minutes. The

5   temperature was then varied between 2920°F and 2976°F for 15 cycles. Each cycle consisted of lowering the temperature continuously over 7 minutes and raising the temperature continuously over 7 minutes. After the 15 cycles were completed, a gas addition lance was lowered into the molten metal to a position approximately 2" from the bottom of the reactor and a 0.15 L/min flow of argon was begun. The

10 temperature of the Silicon was varied over another 5 cycles between 2920°F and 2976°F.

After the fifth cycle, the reactor temperature was lowered to 2900°F over a 10 minute period with continued argon addition. The graphite saturation assemblies were reinstalled in the Silicon and remained there for 1 hour. The graphite

15 saturation assemblies were removed.

The reactor temperature was lowered to 2895°F over 5 minutes. The reactor was held at this temperature for 5 minutes with continued argon addition. The temperature was then varied between 2886°F and 2895°F over 20 cycles. Each cycle consisted of lowering the temperature continuously over 9 minutes and raising

20 the temperature continuously over 9 minutes. The argon addition ceased after completion of the 20 cycles.

The reactor temperature was lowered to 2873°F over 5 minutes. The temperature was varied between 2868°F and 2873°F over 4 ½ cycles. Each cycle consisted of lowering the temperature continuously over 5 minutes and raising the

25 temperature continuously over 3 minutes. In addition, while raising the temperature, a 0.15 L/min flow of argon was added, and while lowering the temperature, a 0.15 L/min flow of nitrogen was added.

The reactor temperature was lowered to 2863°F over 5 minutes. The temperature was varied between 2811°F and 2863°F for 15.5 cycles. Each cycle

30 consisted of lowering the temperature continuously over 15 minutes and raising the temperature continuously over 15 minutes. In addition, while raising the temperature, a 0.15 L/min flow of argon was added, and while lowering the

temperature, a 0.15 L/min flow of nitrogen was added. All gas addition, except for the purge of nitrogen ceased after the 15.5 cycles were completed.

The temperature was varied between 2833°F and 2811°F for one cycle. The cycle consisted of raising the temperature continuously over 15 minutes and

5   lowering the temperature continuously over 15 minutes. The gas addition lance was removed .

The reactor temperature was slowly cooled by lowering the induction furnace power to 1 kW or less s the ingot cooled. After solidification, the Silicon was cooled to approximately ambient temperature in water.

10   ANALYTICAL PROTOCOLS

XRF, grain size, magnetism, and chemical reactivity measurements were carried out by the procedures described in Example 1.

ANALYTICAL RESULTS

An x-ray fluorescence analysis of the silicon sample is provided in Figures

15   45A and 45B, with the K_α and L_α peaks of a silicon control standard shown for reference.

An x-ray fluorescence analysis of the silicon sample is provided in Figure 46A, with the K_α peak of an aluminum control standard shown for reference.

An x-ray fluorescence analysis of the silicon sample is provided in Figure

20   46B, with the K_α peak of a titanium control standard shown for reference.

An x-ray fluorescence analysis of the silicon sample is provided in Figure 47A, with the K_α peak of a sulfur control standard shown for reference.

An x-ray fluorescence analysis of the silicon sample is provided in Figure 47B, with the K_α peak of a chlorine (from potassium chloride) control standard

25   shown for reference.

An x-ray fluorescence analysis of the silicon sample is provided in Figure 48A, with the K_α peak of a gallium control standard shown for reference.

An x-ray fluorescence analysis of the silicon sample is provided in Figure 48B, with the K_α peak of a potassium control standard shown for reference.

Summary data showing the apparent elemental composition of the product of Example 7 is shown in Tables 24-27, as was measured by an XRF analysis using a Uniquant software package. The apparent elemental composition of the product varies by position, which is indicated in each table.

5   No unexpected magnetic activity or chemical reactivity were recorded for the ingot. The manufactured silicon system did appear shiny on its axial (top) face and dull on is radial (side) face. The ingot retained minimal refractory upon removal from the reactor.

EXAMPLE 8

10   EXPERIMENTAL PROCEDURE FOR IRON 15-01-02

A cylindrical alumina-based crucible (99.68% Al_2O_3, 0.07% SiO_2, 0.08% Fe_2O_3, 0.04% CaO, 0.12% Na_2O_3; 4.5" O.D. x 3.75" I.D. x 10" depth) of a 100 pound induction furnace reactor supplied by Inductotherm, fitted with a 75-30R Powertrak power supply was charged with 2000 g Iron (99.98% purity) and 200 g

15   carbon through its charging port. The reactor was fitted with a graphite cap with a ceramic liner (i.e. the crucible, from Engineering Ceramics). During the entire procedure, a slight positive pressure of nitrogen (~0.5 psi) was maintained in the reactor using a continuous backspace purge. The reactor was heated to the metal charge liquidus point plus 300°F, at a rate no greater than 300°F/hour, as limited by

20   the integrity of the crucible. The induction furnace operated in the frequency range of 0 kHz to 3000 kHz, with frequency determined by a temperature-controlled feedback loop implementing an Omega Model CN300 temperature controller. Upon reaching 2800°F, the reactor was charged with an additional 2595 g iron over an hour.

25   The temperature was again increased to 2850°F again using a rate no greater than 300°F/hour. When this temperature was reached, graphite saturation assemblies (3/8" OD, 36" long high purity (<5 ppm impurities) graphite rods) were inserted to the bottom of the iron charge through ports located in the top plate. The iron was held at 2850°F for 4 hours. Every 30 minutes during the hold period, an

30   attempt was made to lower the graphite saturation assemblies as dissolution

occurred. As the iron became saturated with carbon, the graphite saturation assemblies were consumed. After the 4 hour hold period was complete, the graphite saturation assemblies were removed.

The reactor temperature was increased to 3360°F over 7 minutes. The temperature was then varied between 2993°F and 3360°F for 15 cycles. Each cycle consisted of lowering the temperature continuously over 7 minutes and raising the temperature continuously over 7 minutes. After the 15 cycles were completed, a gas addition lance was lowered into the molten metal to a position approximately 2" from the bottom of the reactor and a 0.15 L/min flow of argon was begun. The temperature of the iron was varied over another 5 cycles between 2993°F and 3360°F.

After the fifth cycle, the reactor temperature was lowered to 2850°F over a 10 minute period with continued argon addition. The graphite saturation assemblies were reinstalled in the iron and remained there for 1 hour. The graphite saturation assemblies were removed.

The reactor temperature was lowered to 2819°F over 5 minutes. The reactor was held at this temperature for 5 minutes with continued argon addition. The temperature was then varied between 2622°F and 2818°F over 20 cycles. Each cycle consisted of lowering the temperature continuously over 9 minutes and raising the temperature continuously over 9 minutes. The argon addition ceased after completion of the 20 cycles.

The reactor temperature was lowered to 2724°F over 5 minutes. The temperature was varied between 2622°F and 2724°F over 4 ½ cycles. Each cycle consisted of lowering the temperature continuously over 5 minutes and raising the temperature continuously over 3 minutes. In addition, while raising the temperature, a 0.15 L/min flow of argon was added, and while lowering the temperature, a 0.15 L/min flow of nitrogen was added.

The reactor temperature was lowered to 2586°F over 5 minutes. The temperature was varied between 2133°F and 2586°F for 15.5 cycles. Each cycle consisted of lowering the temperature continuously over 15 minutes and raising the temperature continuously over 15 minutes. In addition, while raising the temperature, a 0.15 L/min flow of argon was added, and while lowering the

temperature, a 0.15 L/min flow of nitrogen was added. All gas addition, except for
the purge of nitrogen ceased after the 15.5 cycles were completed.

The temperature was varied between 2340°F and 2133°F for one cycle. The
cycle consisted of raising the temperature continuously over 15 minutes and
5 lowering the temperature continuously over 15 minutes. The gas addition lance was
removed .

The reactor temperature was cooled by lowering the induction furnace
power to 1 kW or less as the ingot cooled. After solidification, the iron was cooled
to approximately ambient temperature in water.

10 ANALYTICAL PROTOCOLS

XRF, grain size, magnetism, and chemical reactivity measurements were
carried out by the procedures described in Example 1.

ANALYTICAL RESULTS

An x-ray fluorescence analysis of the iron sample is provided in Figures
15 49A and 49B, with the K_α and L_α peaks of an iron control standard shown for
reference.

An x-ray fluorescence analysis of the iron sample is provided in Figure 50A,
with the K_α peak of an aluminum control standard shown for reference.

An x-ray fluorescence analysis of the iron sample is provided in Figure 50B,
20 with the K_α peak of an zirconium control standard shown for reference.

An x-ray fluorescence analysis of the iron sample is provided in Figure 51A,
with the K_α peak of a sulfur control standard shown for reference.

An x-ray fluorescence analysis of the iron sample is provided in Figure 51B,
with the K_α peak of a chlorine (from potassium chloride) control standard shown for
25 reference.

Summary data showing the apparent elemental composition of the product
of Example 8 is shown in Tables 28-29, as was measured by an XRF analysis using
a Uniquant software package. The apparent elemental composition of the product
varies by position, which is indicated in each table.

The manufactured iron exhibited no unexpected magnetic activity. The reactivity relative to that which would be expected from natural iron has not been quantified. The ingot appears glassy or shiny on its axial (top) face and dull on its radial (side) face. The manufactured iron retained a negligible amount of refractory

5   upon removal from the reactor, but cracked upon retrieval. The ingot had no internal voids.

EXAMPLE 9
EXPERIMENTAL PROCEDURE FOR IRON W/VANADIUM, CHROMIUM AND MANGANESE 15-01-03

10   A cylindrical alumina-based crucible (99.68% Al_2O_3, 0.07% SiO_2, 0.08% Fe_2O_3, 0.04% CaO, 0.12% Na_2O_3; 4.5" O.D. x 3.75" I.D. x 10" depth) of a 100 pound induction furnace reactor supplied by Inductotherm, fitted with a 75-30R Powertrak power supply, was charged with 2000 g Iron (99.98% purity), plus 91.9 g each of Vanadium (99.5% pure), Chromium (99% pure), and Manganese (99.9%

15   pure), plus 200 g of carbon through its charging port. The reactor was fitted with a graphite cap with a ceramic liner (i.e., the crucible, from Engineering Ceramics). During the entire procedure, a slight positive pressure of nitrogen (~0.5 psi) was maintained in the reactor using a continuous backspace purge. The reactor was heated to the metal charge liquidus point plus 300°F, at a rate no greater than

20   300°F/hour as limited by the integrity of the crucible. The induction furnace operated in the frequency range of 0 kHz to 3000 kHz, with frequency determined by a temperature-controlled feedback loop implementing an Omega Model CN300 temperature controller. Upon reaching 2800°F, the reactor was charged with an additional 2319.3 g iron over an hour.

25   The temperature was again increased to 2850°F again using a rate no greater than 300°F/hour. When this temperature was reached, graphite saturation assemblies (3/8" OD, 36" long high purity graphite rods) were inserted to the bottom of the metal charge through ports located in the top plate . The alloy was held at 2850°F for 4 hours. Every 30 minutes during the hold period, an attempt

30   was made to lower the graphite saturation assemblies as dissolution occurred. As

the alloy became saturated with carbon, the graphite saturation assemblies were consumed. After the 4 hour hold period was complete, the graphite saturation assemblies were removed.

The reactor temperature was increased to 3360°F over 7 minutes. The temperature was then varied between 2993°F and 3360°F for 15 cycles. Each cycle consisted of lowering the temperature continuously over 7 minutes and raising the temperature continuously over 7 minutes. After the 15 cycles were completed, a gas addition lance was lowered into the molten metal to a position approximately 2" from the bottom of the reactor and a 0.15 L/min flow of argon was begun. The temperature of the iron was varied over another 5 cycles between 2993°F and 3360°F.

After the fifth cycle, the reactor temperature was lowered to 2850°F over a 10 minute period with continued argon addition. The graphite saturation assemblies were reinstalled in the iron and remained there for 1 hour. The graphite saturation assemblies were removed.

The reactor temperature was lowered to 2819°F over 5 minutes. The reactor was held at this temperature for 5 minutes with continued argon addition. The temperature was then varied between 2622°F and 2818°F over 20 cycles. Each cycle consisted of lowering the temperature continuously over 9 minutes and raising the temperature continuously over 9 minutes. The argon addition ceased after completion of the 20 cycles.

The reactor temperature was lowered to 2724°F over 5 minutes. The temperature was varied between 2622°F and 2724°F over 4 ½ cycles. Each cycle consisted of lowering the temperature continuously over 5 minutes and raising the temperature continuously over 3 minutes. In addition, while raising the temperature, a 0.15 L/min flow of argon was added, and while lowering the temperature, a 0.15 L/min flow of nitrogen was added.

The reactor temperature was lowered to 2586°F over 5 minutes. The temperature was varied between 2133°F and 2586°F for 15.5 cycles. Each cycle consisted of lowering the temperature continuously over 15 minutes and raising the temperature continuously over 15 minutes. In addition, while raising the temperature, a 0.15 L/min flow of argon was added, and while lowering the

temperature, a 0.15 L/min flow of nitrogen was added. All gas addition, except for the purge of nitrogen ceased after the 15.5 cycles were completed.

The temperature was varied between 2340°F and 2133°F for one cycle. The cycle consisted of raising the temperature continuously over 15 minutes and
5   lowering the temperature continuously over 15 minutes. The gas addition lance was removed.

The reactor temperature was cooled by lowering the induction furnace power to 1 kW or less as the ingot cooled. The iron/vanadium/chromium/manganese alloy was the cooled to approximately
10   ambient temperature in water.

ANALYTICAL PROTOCOLS

XRF, grain size, magnetism, and chemical reactivity measurements were carried out by the procedures described in Example 1.

ANALYTICAL RESULTS
15   An x-ray fluorescence analysis of the iron/vanadium/chromium/manganese alloy sample is provided in Figure 52, with the K_α peak of a chromium (from chromium(III) oxide) control standard shown for reference.

An x-ray fluorescence analysis of the iron/vanadium/chromium/manganese alloy sample is provided in Figure 53, with the K_α peak of an iron control standard
20   shown for reference.

An x-ray fluorescence analysis of the iron/vanadium/chromium/manganese alloy sample is provided in Figure 54, with the K_α peak of a vanadium control standard shown for reference.

An x-ray fluorescence analysis of the iron/vanadium/chromium/manganese
25   alloy sample is provided in Figure 55, with the K_α peak of a manganese control standard shown for reference.

An x-ray fluorescence analysis of the iron/vanadium/chromium/manganese alloy sample is provided in Figure 56, in the region of the K_α peak of a sulfur control standard.

Summary data showing the apparent elemental composition of the product of Example 9 is shown in Tables 40-41, as was measured by an XRF analysis using a Uniquant software package. The apparent elemental composition of the product varies by position, which is indicated in each table.

5   Upon polishing the manufactured alloy in preparation for XRF analysis, the alloy was noted to be particularly hard and a stratification pattern that was not attributable to the polishing materials was exposed. The relative hardness of the alloy was tested and the Moh's hardness was found to be greater than what would be expected from a natural alloy of a similar composition. The radial surface of the

10   ingot had a shiny or glassy appearance, while the axial surface appeared dull, thus reflecting bulk anisotropic behavior.

The manufactured alloy had no unexpected magnetic activity. The ingot retained a negligible amount of refractory upon retrieval from the reactor, but did crack.

15   EXAMPLE 10
EXPERIMENTAL PROCEDURE FOR NICKEL W/TANTALUM, HAFNIUM AND TUNGSTEN 15-01-04

A cylindrical alumina-based crucible (99.68% Al_2O_3, 0.07% SiO_2, 0.08% Fe_2O_3, 0.04% CaO, 0.12% Na_2O_3; 4.5" O.D. x 3.75" I.D. x 10" depth) of a 100

20   pound induction furnace reactor supplied by Inductotherm, fitted with a 75-30R Powertrak power supply and was charged with 2500 g Nickel (99.9% purity), plus 100 g each of Hafnium (99.9% pure), W (99.9% pure), Ta (99.98% pure), and carbon through its charging port. The reactor was fitted with a graphite cap with a ceramic liner (i.e., the crucible, from Engineering Ceramics). During the entire

25   procedure, a slight positive pressure of nitrogen (~0.5 psi) was maintained in the reactor using a continuous backspace purge. The reactor was heated to the metal charge liquidus point plus 300°F, at a rate no greater than 300°F/hour, as limited by the integrity of the crucible. The induction furnace operated in the frequency range of 0 kHz to 3000 kHz, with frequency determined by a temperature-controlled

30   feedback loop implementing an Omega Model CN300 temperature controller.

Upon reaching 2800°F, the reactor was charged with an additional 2200 g nickel over an hour.

The temperature was again increased to 2850°F again using a rate no greater than 300°F/hour. When this temperature was reached, graphite saturation assemblies (3/8" OD, 36" long high purity (<5 ppm impurities) graphite rods) were inserted to the bottom of the metal charge through ports located in the top plate. The alloy was held at 2850°F for 4 hours. Every 30 minutes during the hold period, an attempt was made to lower the graphite saturation assemblies as dissolution occurred. As the alloy became saturated with carbon, the graphite saturation assemblies were consumed. After the 4 hour hold period was complete, the graphite saturation assemblies were removed.

The reactor temperature was increased to 3256°F over 7 minutes. The temperature was then varied between 2950°F and 3256°F for 15 cycles. Each cycle consisted of lowering the temperature continuously over 7 minutes and raising the temperature continuously over 7 minutes. After the 15 cycles were completed, a gas addition lance was lowered into the molten metal to a position approximately 2" from the bottom of the reactor and a 0.15 L/min flow of argon was begun. The temperature of the alloy was varied over another 5 cycles between 2950°F and 3256°F.

After the fifth cycle, the reactor temperature was lowered to 2850°F over a 10 minute period with continued argon addition. The graphite saturation assemblies were reinstalled in the alloy and remained there for 1 hour. The graphite saturation assemblies were removed.

The reactor temperature was lowered to 2829°F over 5 minutes. The reactor was held at this temperature for 5 minutes with continued argon addition. The temperature was then varied between 2790°F and 2829°F over 20 cycles. Each cycle consisted of lowering the temperature continuously over 9 minutes and raising the temperature continuously over 9 minutes. The argon addition ceased after completion of the 20 cycles.

The reactor temperature was lowered to 2770°F over 5 minutes. The temperature was varied between 2710°F and 2770°F over 4 ½ cycles. Each cycle consisted of lowering the temperature continuously over 5 minutes and raising the

temperature continuously over 3 minutes. In addition, while raising the temperature, a 0.15 L/min flow of argon was added, and while lowering the temperature, a 0.15 L/min flow of nitrogen was added.

The reactor temperature was lowered to 2691°F over 5 minutes. The
5 temperature was varied between 2492°F and 2691°F for 15.5 cycles. Each cycle consisted of lowering the temperature continuously over 15 minutes and raising the temperature continuously over 15 minutes. In addition, while raising the temperature, a 0.15 L/min flow of argon was added, and while lowering the temperature, a 0.15 L/min flow of nitrogen was added. All gas addition, except for
10 the purge of nitrogen ceased after the 15.5 cycles were completed.

The temperature was varied between 2571°F and 2492°F for one cycle. The cycle consisted of raising the temperature continuously over 15 minutes and lowering the temperature continuously over 15 minutes. The gas addition lance was removed.

15 The reactor temperature was slowly cooled by lowering the induction furnace power to 1 kW or less as the ingot cooled. After solidification, the alloy was cooled to approximately ambient temperature in water.

ANALYTICAL PROTOCOLS

XRF, grain size, magnetism, and chemical reactivity measurements were
20 carried out by the procedures described in Example 1.

ANALYTICAL RESULTS

An x-ray fluorescence analysis of the nickel/hafnium/tantalum/tungsten alloy sample is provided in Figure 57, with the K_α peak of a tantalum control standard shown for reference.
25 An x-ray fluorescence analysis of the nickel/hafnium/tantalum/tungsten alloy sample is provided in Figure 58, with the K_α peak of a tungsten control standard shown for reference.

An x-ray fluorescence analysis of the nickel/hafnium/tantalum/tungsten alloy sample is provided in Figure 59, with the K_α peak of a hafnium control
30 standard shown for reference.

An x-ray fluorescence analysis of the nickel/hafnium/tantalum/tungsten alloy sample is provided in Figure 60, in the region of the K_α peak of a sulfur control standard.

An x-ray fluorescence analysis of the nickel/hafnium/tantalum/tungsten
5   alloy sample is provided in Figure 61, with the K_α peak of a nickel control standard shown for reference.

Summary data showing the apparent elemental composition of the product of Example 10 is shown in Tables 30-31, as was measured by an XRF analysis using a Uniquant software package. The apparent elemental composition of the
10   product varies by position, which is indicated in each table.

The manufactured nickel-based alloy exhibited clear anisotropic behavior with respect to color and reactivity similar to the anisotropy observed via XRF. The sides were covered with a large amount of refractory retained after the ingot was retrieved from the reactor. The top face had a classic metallic sheen. No
15   unexpected magnetic activity was observed. The ingot did not upon removal from the reactor or exhibit any internal voids.

EXAMPLE 11
EXPERIMENTAL PROCEDURE FOR COPPER 14-00-01

A cylindrical alumina-based crucible (89.07% Al_2O_3, 10.37% SiO_2, 0.16%
20   TiO_2, 0.15% Fe_2O_3, 0.03% CaO, 0.01% MgO, 0.02% Na_2O_3, 0.02% K_2O; 9" O.D. x 7.75" I.D. x 14" depth) of a 100 pound induction furnace reactor supplied by Inductotherm, fitted with a 75-30R Powertrak power supply, was charged with 100 pounds copper (99.98% purity) through its charging port. During the entire procedure, a slight positive pressure of nitrogen (~0.5 psi) was maintained in the
25   reactor using a continuous backspace purge. The reactor was heated to the metal charge liquidus point plus 300°F, at a rate no greater than 300°F/hour, as limited by the integrity of the crucible. The induction furnace operated in the frequence range of 0 kHz to 3000 kHz, with frequency determined by a temperature-controlled feedback loop implementing an Omega Model CN300 temperature controller.

The temperature was again increased to 2462°F again using a rate no greater than 300°F/hour. When this temperature was reached, graphite saturation assemblies (3/8" OD, 36" long high purity (<5 ppm impurities) graphite rods) were inserted to the bottom of the copper charge through ports located in the top plate.

5   The copper was held at 2462°F for 4 hours. Every 30 minutes during the hold period, an attempt was made to lower the graphite saturation assemblies as dissolution occurred. As the copper became saturated with carbon, the graphite saturation assemblies were consumed. After the 4 hour hold period was complete, the graphite saturation assemblies were removed.

10   The reactor temperature was increased to 2480°F over 7 minutes. The temperature was then varied between 2480°F and 2530°F for 15 cycles. Each cycle consisted of raising the temperature continuously over 7 minutes and lowering the temperature continuously over 7 minutes. After the 15 cycles were completed, a gas addition lance was lowered into the molten metal to a position approximately 2"

15   from the bottom of the reactor and a 1.5 L/min flow of argon was begun. The temperature of the copper was varied over another 5 cycles between 2480°F and 2530°F.

After the fifth cycle, the reactor temperature was lowered to 2462°F over a 10 minute period with continued argon addition. The graphite saturation assemblies

20   were reinstalled in the copper and remained there for 1 hour. The graphite saturation assemblies were removed.

The reactor temperature was lowered to 2459°F over 5 minutes. The reactor was held at this temperature for 5 minutes with continued argon addition. The temperature was then varied between 2459°F and 2453°F over 20 cycles. Each

25   cycle consisted of lowering the temperature continuously over 9 minutes and raising the temperature continuously over 9 minutes. The argon addition ceased after completion of the 20 cycles.

The reactor temperature was lowered to 2450°F over 5 minutes. The temperature was varied between 2450°F and 2441°F over 4 ½ cycles. Each cycle

30   consisted of lowering the temperature continuously over 5 minutes and raising the temperature continuously over 3 minutes. In addition, while raising the temperature,

a 1.5 L/min flow of argon was added, and while lowering the temperature, a 1.5 L/min flow of nitrogen was added.

The reactor temperature was lowered to 2438°F over 5 minutes. The temperature was varied between 2438°F and 2406°F for 15.5 cycles. Each cycle consisted of lowering the temperature continuously over 15 minutes and raising the temperature continuously over 15 minutes. In addition, while raising the temperature, a 1.5 L/min flow of argon was added, and while lowering the temperature, a 1.5 L/min flow of nitrogen was added. All gas addition, except for the purge of nitrogen ceased after the 15.5 cycles were completed.

The temperature was varied between 2406°F and 2419°F for one cycle. The cycle consisted of raising the temperature continuously over 15 minutes and lowering the temperature continuously over 15 minutes. The gas addition lance was removed.

The reactor temperature was rapidly cooled by quenching in water, so that the copper solidified into an ingot.

ANALYTICAL PROTOCOLS

XRF, grain size, magnetism, and chemical reactivity measurements were carried out by the procedures described in Example 1.

ANALYTICAL RESULTS

Summary data showing the apparent elemental composition of the product of Example 11 is shown in Tables 32-33, as was measured by an XRF analysis using a Uniquant software package. The apparent elemental composition of the product varies by position, which is indicated in each table.

Immediately after the method described above was completed, multiple discrete magnetic spots attracted by a 1/8" diameter neodymium iron boron magnet were observed in a sinusoidal pattern. The ingot exhibited point attraction to iron filings at reduced temperatures at or near 77 K. Over days to months, the strength of the magnetic attraction decreased on a fraction of the locations exhibiting magnetic attraction or attraction to iron filings.

Various forms of ligated chlorine (e.g., HCl and MCl, where M is a metal as defined above) readily reacted with the manufactured copper form yielding product distributions distinguishable from natural copper, thereby demonstrating a change in chemical reactivity. This reactivity increased over time.

5 Extremely large grain sizes (i.e., greater than 1") were observed, which is uncharacteristic and previously unreported in natural copper systems (typically, copper grains sizes are 10-100 μm). Unique changes in coloration were observed with the crossing of grain boundaries; however, the overall coloration mimicked natural copper.

10 EXAMPLE 12

EXPERIMENTAL PROCEDURE FOR COPPER 14-00-03

A cylindrical alumina-based crucible (89.07% Al_2O_3, 10.37% SiO_2, 0.16% TiO_2, 0.15% Fe_2O_3, 0.03% CaO, 0.01% MgO, 0.02% Na_2O_3, 0.02% K_2O; 9" O.D. x 7.75" I.D. x 14" depth) of a 100 pound induction furnace reactor supplied by

15 Inductotherm, fitted with a 75-30R Powertrak power supply and was charged with 100 pounds copper (99.98% purity) through its charging port. During the entire procedure, a slight positive pressure of nitrogen (~0.5 psi) was maintained in the reactor using a continuous backspace purge. The reactor was heated to the metal charge liquidus point plus 300°F, at a rate no greater than 300°F/hour, as limited by

20 the integrity of the crucible. The induction furnace operated in the frequence range of 0 kHz to 3000 kHz, with frequency determined by a temperature-controlled feedback loop implementing an Omega Model CN300 temperature controller.

The temperature was again increased to 2462°F again using a rate no greater than 300˚F/hour. When this temperature was reached, graphite saturation

25 assemblies (3/8" OD, 36" long high purity (<5 ppm impurities) graphite rods) were inserted to the bottom of the copper charge through ports located in the top plate. The copper was held at 2462°F for 4 hours. Every 30 minutes during the hold period, an attempt was made to lower the graphite saturation assemblies as dissolution occurred. As the copper became saturated with carbon, the graphite

saturation assemblies were consumed. After the 4 hour hold period was complete, the graphite saturation assemblies were removed.

The reactor temperature was increased to 2480°F over 7 minutes. The temperature was then varied between 2480°F and 2530°F for 15 cycles. Each cycle consisted of raising the temperature continuously over 7 minutes and lowering the temperature continuously over 7 minutes. After the 15 cycles were completed, a gas addition lance was lowered into the molten metal to a position approximately 2" from the bottom of the reactor and a 1.5 L/min flow of argon was begun.. The temperature of the copper was varied over another 5 cycles between 2480°F and 2530°F.

After the fifth cycle, the reactor temperature was lowered to 2462°F over a 10 minute period with continued argon addition. The graphite saturation assemblies were reinstalled in the copper and remained there for 1 hour. The graphite saturation assemblies were removed.

The reactor temperature was lowered to 2459°F over 5 minutes. The reactor was held at this temperature for 5 minutes with continued argon addition. The temperature was then varied between 2459°F and 2453°F over 20 cycles. Each cycle consisted of lowering the temperature continuously over 9 minutes and raising the temperature continuously over 9 minutes. The argon addition ceased after completion of the 20 cycles.

The reactor temperature was lowered to 2450°F over 5 minutes. The temperature was varied between 2450°F and 2441°F over 4 ½ cycles. Each cycle consisted of lowering the temperature continuously over 5 minutes and raising the temperature continuously over 3 minutes. In addition, while raising the temperature, a 1.5 L/min flow of argon was added, and while lowering the temperature, a 1.5 L/min flow of nitrogen was added.

The reactor temperature was lowered to 2438°F over 5 minutes. The temperature was varied between 2438°F and 2406°F for 15.5 cycles. Each cycle consisted of lowering the temperature continuously over 15 minutes and raising the temperature continuously over 15 minutes. In addition, while raising the temperature, a 1.5 L/min flow of argon was added, and while lowering the

temperature, a 1.5 L/min flow of nitrogen was added. All gas addition, except for the purge of nitrogen ceased after the 15.5 cycles were completed.

The temperature was varied between 2406°F and 2419°F for one cycle. The cycle consisted of raising the temperature continuously over 15 minutes and

5   lowering the temperature continuously over 15 minutes. The gas addition lance was removed.

The reactor temperature was rapidly cooled by quenching in water, so that the copper solidified into an ingot.

ANALYTICAL PROTOCOLS

10   XRF, grain size, magnetism, and chemical reactivity measurements were carried out by the procedures described in Example 1.

ANALYTICAL RESULTS

Summary data showing the apparent elemental composition of the product of Example 12 is shown in Tables 34-35, as was measured by an XRF analysis

15   using a Uniquant software package. The apparent elemental composition of the product varies by position, which is indicated in each table.

This manufactured copper system demonstrated an ability to change color (i.e., visible light spectrum emission) dependent upon electromagnetic stimulation. The color of the top, glassy surface of an ingot changed under different lighting

20   conditions. While the (radial) side of the ingot was a matte pink color, the top of the ingot (axial face) has a glassy color, which can vary from intense burgundy to golden bronze to burnished orange. These differences in appearance reflect the anisotropy detected via the XRFs.

The radial surface of the ingot was covered with magnetically active spots.

25   The magnetism of the ingot decreased over time. Altered chemical reactivity, particularly with respect to ligated chlorine, was observed on axial surfaces. The chemical reactivity increased over time. Radial surfaces appeared unaffected and were free from refractory (material from the crucible).

EXAMPLE 13

EXPERIMENTAL PROCEDURE FOR COPPER 14-00-04

A cylindrical alumina-based crucible (89.07% Al_2O_3, 10.37% SiO_2, 0.16% TiO_2, 0.15% Fe_2O_3, 0.03% CaO, 0.01% MgO, 0.02% Na_2O_3, 0.02% K_2O; 9" O.D. x

5   7.75" I.D. x 14" depth) of a 100 pound induction furnace reactor supplied by Inductotherm, fitted with a 75-30R Powertrak power supply was charged with 100 pounds copper (99.98% purity) through its charging port. During the entire procedure, a slight positive pressure of nitrogen (~0.5 psi) was maintained in the reactor using a continuous backspace purge. The reactor was heated to the metal

10   charge liquidus point plus 300°F, at a rate no greater than 300°F/hour, as limited by the integrity of the crucible. The induction furnace operated in the frequence range of 0 kHz to 3000 kHz, with frequency determined by a temperature-controlled feedback loop implementing an Omega Model CN300 temperature controller

The temperature was again increased to 2462°F again using a rate no greater

15   than 300°F/hour. When this temperature was reached, graphite saturation assemblies (3/8" OD, 36" long high purity (<5 ppm impurities) graphite rods) were inserted to the bottom of the copper charge through ports located in the top plate. The copper was held at 2462°F for 4 hours. Every 30 minutes during the hold period, an attempt was made to lower the graphite saturation assemblies as

20   dissolution occurred. As the copper became saturated with carbon, the graphite saturation assemblies were consumed. After the 4 hour hold period was complete, the graphite saturation assemblies were removed.

The reactor temperature was increased to 2480°F over 7 minutes. The temperature was then varied between 2480°F and 2530°F for 15 cycles. Each cycle

25   consisted of raising the temperature continuously over 7 minutes and lowering the temperature continuously over 7 minutes. After the 15 cycles were completed, a gas addition lance was lowered into the molten metal to a position approximately 2" from the bottom of the reactor and a 1.5 L/min flow of argon was begun The temperature of the copper was varied over another 5 cycles between 2480°F and

30   2530°F.

After the fifth cycle, the reactor temperature was lowered to 2462°F over a 10 minute period with continued argon addition. The graphite saturation assemblies were reinstalled in the copper and remained there for 1 hour. The graphite saturation assemblies were removed.

5   The reactor temperature was lowered to 2459°F over 5 minutes. The reactor was held at this temperature for 5 minutes with continued argon addition. The temperature was then varied between 2459°F and 2453°F over 20 cycles. Each cycle consisted of lowering the temperature continuously over 9 minutes and raising the temperature continuously over 9 minutes. The argon addition ceased

10   after completion of the 20 cycles.

The reactor temperature was lowered to 2450°F over 5 minutes. The temperature was varied between 2450°F and 2441°F over 4 ½ cycles. Each cycle consisted of lowering the temperature continuously over 5 minutes and raising the temperature continuously over 3 minutes. In addition, while raising the temperature,

15   a 1.5 L/min flow of argon was added, and while lowering the temperature, a 1.5 L/min flow of nitrogen was added.

The reactor temperature was lowered to 2438°F over 5 minutes. The temperature was varied between 2438°F and 2406°F for 15.5 cycles. Each cycle consisted of lowering the temperature continuously over 15 minutes and raising the

20   temperature continuously over 15 minutes. In addition, while raising the temperature, a 1.5 L/min flow of argon was added, and while lowering the temperature, a 1.5 L/min flow of nitrogen was added. All gas addition, except for the purge of nitrogen ceased after the 15.5 cycles were completed.

The temperature was varied between 2406°F and 2419°F for one cycle. The

25   cycle consisted of raising the temperature continuously over 15 minutes and lowering the temperature continuously over 15 minutes. The gas addition lance was removed.

The reactor temperature was slowly cooled and was subsequently quenched in water, so that the copper solidified into an ingot.

ANALYTICAL PROTOCOLS

XRF, grain size, magnetism, and chemical reactivity measurements were carried out by the procedures described in Example 1.

ANALYTICAL RESULTS

5   Summary data showing the apparent elemental composition of the product of Example 13 is shown in Tables 36-37, as was measured by an XRF analysis using a Uniquant software package. The apparent elemental composition of the product varies by position, which is indicated in each table.

The manufactured copper ingot exhibited many of the characteristic patterns

10   observed in previous examples. Differences in the coloration and appearance of the axial and radial directions were observed: matte, burgundy brown coloration on the side, and glassy on the top with demarcations.

This ingot had internal voids. The ingot demonstrated enhanced chemical reactivity on the axial surfaces. Refractory was found to be tightly bound to select

15   portions of the radial surfaces. Minimal magnetic activity was detected.

EXAMPLE 14
EXPERIMENTAL PROCEDURE FOR COPPER 15-00-01

A cylindrical alumina-based crucible (89.07% Al_2O_3, 10.37% SiO_2, 0.16% TiO_2, 0.15% Fe_2O_3, 0.03% CaO, 0.01% MgO, 0.02% Na_2O_3, 0.02% K_2O; 9" O.D. x

20   7.75" I.D. x 14" depth) of a 100 pound induction furnace reactor supplied by Inductotherm, fitted with a 75-30R Powertrak power supply and was charged with 100 pounds copper (99.98% purity) through its charging port. During the entire procedure, a slight positive pressure of nitrogen (~0.5 psi) was maintained in the reactor using a continuous backspace purge. The reactor was heated to the metal

25   charge liquidus point plus 300°F, at a rate no greater than 300°F/hour, as limited by the integrity of the crucible. The induction furnace operated in the frequence range of 0 kHz to 3000 kHz, with frequency determined by a temperature-controlled feedback loop implementing an Omega Model CN300 temperature controller.

The temperature was again increased to 2462°F again using a rate no greater than 300°F/hour. When this temperature was reached, graphite saturation assemblies (3/8" OD, 36" long high purity (<5 ppm impurities) graphite rods) were inserted to the bottom of the copper charge through ports located in the top plate.

5   The copper was held at 2462°F for 4 hours. Every 30 minutes during the hold period, an attempt was made to lower the graphite saturation assemblies as dissolution occurred. As the copper became saturated with carbon, the graphite saturation assemblies were consumed. After the 4 hour hold period was complete, the graphite saturation assemblies were removed.

10   The reactor temperature was increased to 2480°F over 7 minutes. The temperature was then varied between 2480°F and 2530°F for 15 cycles. Each cycle consisted of raising the temperature continuously over 7 minutes and lowering the temperature continuously over 7 minutes. After the 15 cycles were completed, a gas addition lance was lowered into the molten metal to a position approximately 2"

15   from the bottom of the reactor and a 1.5 L/min flow of argon was begun. The temperature of the copper was varied over another 5 cycles between 2480°F and 2530°F.

The after the fifth cycle, the reactor temperature was lowered to 2462°F over a 10 minute period with continued argon addition. The graphite saturation assemblies

20   were reinstalled in the copper and remained there for 1 hour. The graphite saturation assemblies were removed.

The reactor temperature was lowered to 2459°F over 5 minutes. The reactor was held at this temperature for 5 minutes with continued argon addition. The temperature was then varied between 2459°F and 2453°F over 20 cycles. Each

25   cycle consisted of lowering the temperature continuously over 9 minutes and raising the temperature continuously over 9 minutes. The argon addition ceased after completion of the 20 cycles.

The reactor temperature was lowered to 2450°F over 5 minutes. The temperature was varied between 2450°F and 2441°F over 4 ½ cycles. Each cycle

30   consisted of lowering the temperature continuously over 5 minutes and raising the temperature continuously over 3 minutes. In addition, while raising the temperature,

a 1.5 L/min flow of argon was added, and while lowering the temperature, a 1.5 L/min flow of nitrogen was added.

The reactor temperature was lowered to 2438°F over 5 minutes. The temperature was varied between 2438°F and 2406°F for 15.5 cycles. Each cycle 5 consisted of lowering the temperature continuously over 15 minutes and raising the temperature continuously over 15 minutes. In addition, while raising the temperature, a 1.5 L/min flow of argon was added, and while lowering the temperature, a 1.5 L/min flow of nitrogen was added. All gas addition, except for the purge of nitrogen ceased after the 15.5 cycles were completed.

10 The temperature was varied between 2406°F and 2419°F for one cycle. The cycle consisted of raising the temperature continuously over 15 minutes and lowering the temperature continuously over 15 minutes. The gas addition lance was removed.

The reactor temperature was slowly cooled and was subsequently quenched 15 in water, so that the copper solidified into an ingot.

ANALYTICAL PROTOCOLS

XRF, grain size, magnetism, and chemical reactivity measurements were carried out by the procedures described in Example 1.

ANALYTICAL RESULTS

20 Summary data showing the apparent elemental composition of the product of Example 14 is shown in Tables 38-39, as was measured by an XRF analysis using a Uniquant software package. The apparent elemental composition of the product varies by position, which is indicated in each table.

The manufactured copper demonstrated an ability to change color (i.e., 25 visible light spectrum emission), dependant upon electromagnetic stimulation. The top (axial face) of the ingot can vary from intense burgundy to a deep golden orange. Additionally, the appearance and color of this ingot reflect the anisotropy detected via the XRF scans. The radial (side) face appears like burnished copper, while the axial (top) face has a glassy appearance.

On the bottom and side faces, each of the grain boundaries is clearly delineated. Each of the grains appears to have a different color, giving the exterior of the ingot an iridescent appears. The ingot did not have an internal void, as ingots of previous examples did. Additionally, the ingot did not exhibit the extensive

5   magnetic activity observed in Examples 11 and 12. The ingot retained an extensive amount of refractory upon retrieval from the reactor.

While this invention has been particularly shown and described with references to preferred embodiments thereof, it will be understood by those skilled in the art that various changes in form and details may be made therein without departing from the

10   scope of the invention encompassed by the appended claims.

Table 1

ANALYSIS REPORT   by Uniquant

```
Spectrometers configuration: ARL 8410 Rh 60kV LiF220 LiF420 Gelll T1AP
Sample ident   =  Tailored - 3
Further info   =
Kappa list     =  15-Nov-94    Channel list =  23-Sep-99
Calculated as :  Elements     Spectral impurity data : CAL.209Teflon
X-ray path     =  Vacuum       Film type    = No supporting film
Case number    =  0   Known Area, %Rest, Diluent/Sample and Mass/Area
Eff.Diam.      = 25.00 mm       Eff.Area     = 490.6 mm2
KnownConc      =  0    %
Rest           =  0    %
Dil/Sample     =  0
Viewed Mass    = 18000.00 mg
Sample Height =   5   mm
```

         < means that the concentration is < 10 ppm

         <2e means wt% < 2 StdErr. The + in Z+El means involved in Sum=100%

Z	wt%	StdErr	Z	wt%	StdErr	Z	wt%	StdErr
SumBe..F	0	0.037	29+Cu	99.58	0.03	51 Sb	<	
11 Na	<		30+Zn	<2e	0.006	52 Te	<	
12 Mg	<		31+Ga	0.012	0.003	53 I	<	
13+Al	0.027	0.012	32 Ge	<2e	0.003	55 Cs	<	
14 Si	<		33 As	<		56 Ba	<	
15+P	0.003	0.001	34 Se	<		SumLa..Lu	0.15	0.07
16+S	0.038	0.003	35 Br	<		72+Hf	<2e	0.022
16+So	0.022	0.003	37 Rb	<		73+Ta	<2e	0.071
17+Cl	0.037	0.003	38 Sr	<		74 W	<	
18+Ar	0.017	0.002	39 Y	<		75 Re	<	
19+K	<2e	0.0008	40 Zr	<		76 Os	<	
20+Ca	0.0050	0.0008	41 Nb	<		77+Ir	0.029	0.008
21 Sc	<		42 Mo	<		78+Pt	0.017	0.007
22+Ti	0.015	0.001	44 Ru	<		79 Au	<2e	0.007
23 V	<		45 Rh	<		80 Hg	<	
24 Cr	<		46 Pd	<		81 Tl	<	
25+Mn	0.0069	0.0007	47 Ag	<		82 Pb	<	
26+Fe	0.015	0.001	48+Cd	0.004	0.002	83 Bi	<	
27 Co	<		49 In	<		90 Th	<2e	0.003
28 Ni	<		50 Sn	<		92 U	<	

==== Light Elements ====	==== Noble Elements =====	==== Lanthanides =======
4 Be	44 Ru <	57+La 0.073 0.006
5 B	45 Rh <	58 Ce <
6 C	46 Pd <	59 Pr <
7 N	47 Ag <	60 Nd <
8 O	75 Re <	62+Sm 0.022 0.003
9 F <	76 Os <	63+Eu 0.015 0.002
	77+Ir 0.029 0.008	64+Gd 0.009 0.002
	78+Pt 0.017 0.007	65+Tb 0.015 0.002
	79 Au <2e 0.007	66 Dy <
		67+Ho <2e 0.013
		68 Er <2e 0.004
		69 Tm <
		70 Yb <2e 0.005
		71+Lu <2e 0.013

```
KnownConc= 0              REST= 0                    D/S= 0
Sum Conc's before normalisation to 100% :   90.6 %
```

Table 2

```
ANALYSIS REPORT                              by Uniquant
----------------------------------------------------------------------------

Spectromet rs configuration:  ARL 8410 Rh 60kV LiF220 LiF420 Ge111 T1AP
Sample ident   =  Tailored - 2
Further info   =
Kappa list     =  15-Nov-94    Channel list  =  23-Sep-99
Calculated as :  Elements      Spectral impurity data : CAL.209Teflon
X-ray path     =  Vacuum        Film type     = No supporting film
Case number    =  0   Known Area, %Rest, Diluent/Sample and Mass/Area
Eff.Diam.      =  25.00 mm      Eff.Area      = 490.6 mm2
KnownConc      =  0    %
Rest           =  0    %
Dil/Sample     =  0
Viewed Mass    =  18000.00 mg
Sample Height  =  5   mm
               <    means that the concentration is <  10 ppm
               <2e  means wt% < 2 StdErr. The + in Z+El means involved in Sum=100%
```

Z	wt%	StdErr	Z	wt%	StdErr	Z	wt%	StdErr
SumBe..F	0	0.036	29+Cu	98.62	0.06	51 Sb	<	
11+Na	0.11	0.02	30+Zn	<2e	0.006	52 Te	<	
12 Mg	<		31+Ga	0.007	0.003	53 I	<	
13+Al	0.44	0.03	32 Ge	<2e	0.003	55 Cs	<	
14+Si	0.107	0.008	33 As	<		56 Ba	<	
15+P	0.004	0.001	34 Se	<		SumLa..Lu	0.087	0.072
16+S	0.19	0.01	35 Br	<		72+Hf	<	
16+So	0.12	0.01	37 Rb	<		73+Ta	<2e	0.071
17+Cl	0.17	0.01	38 Sr	<		74 W	<	
18+Ar	0.027	0.002	39 Y	<		75 Re	<	
19+K	0.0065	0.0010	40 Zr	<		76 Os	<	
20+Ca	0.013	0.001	41 Nb	<		77 Ir	<	
21 Sc	<		42 Mo	<		78+Pt	0.022	0.008
22+Ti	0.015	0.001	44 Ru	<		79 Au	<2e	0.007
23 V	<		45 Rh	<		80 Hg	<	
24 Cr	<		46 Pd	<		81 Tl	<	
25 Mn	<2e	0.0006	47 Ag	<		82 Pb	<	
26+Fe	0.044	0.004	48 Cd	<2e	0.002	83 Bi	<	
27 Co	<		49 In	<		90 Th	<	
28 Ni	<		50 Sn	<		92 U	<	

```
==== Light Elements =====   ==== Noble Elements =====   ==== Lanthanides =======
 4  Be                       44  Ru     <                57+La     0.059    0.005
 5  B                        45  Rh     <                58  Ce    <2e       0.003
 6  C                        46  Pd     <                59  Pr    <2e       0.003
 7  N                        47  Ag     <                60  Nd    <2e       0.002
 8  O                        75  Re     <                62  Sm    <2e       0.003
 9  F       <                76  Os     <                63  Eu    <
                             77  Ir     <                64  Gd    <
                             78+Pt     0.022    0.008    65+Tb     0.005    0.002
                             79  Au    <2e       0.007   66  Dy    <2e       0.008
                                                         67+Ho    <2e       0.015
                                                         68  Er    <2e       0.004
                                                         69  Tm    <
                                                         70  Yb    <
                                                         71+Lu    <

KnownConc= 0                     REST= 0              D/S= 0
Sum Conc's before normalisation to 100% :    96.0 %
```

Table 3

```
                    ANALYSIS REPORT                    by UniQuant
-----------------------------------------------------------------------------

Spectrometers configuration:  ARL 8410 Rh 60kV LiF220 LiF420 Ge111 T1AP
Sample ident   =   Tailored - 1
Further info   =
Kappa list     =  15-Nov-94    Channel list =  20-Jul-94
Calculated as :   Elements.    Spectral impurity data : CAL.209Teflon
X-ray path     =  Vacuum       Film type    = No supporting film
Case number    =  0   Known Area, %Rest, Diluent/Sample and Mass/Area
Eff.Diam.      = 25.00 mm       Eff.Area     = 490.6 mm2
KnownConc      =  0     %
Rest           =  0     %
Dil/Sample     =  0
Viewed Mass    = 18000.00 mg
Sample Height  =  5    mm
                    <   means that the concentration is <  10 ppm
                    <2e means that  Conc < 2 x StdErr
```

Z	wt%	StdErr	Z	wt%	StdErr	Z	wt%	StdErr
SumBe..F	0	0.036	29 Cu	99.84	0.03	51 Sb	<	
11 Na	<		30 Zn	<		52 Te	<	
12 Mg	<		31 Ga	0.007	0.003	53 I	<	
13 Al	<		32 Ge	<2e	0.002	55 Cs	<	
14 Si	<		33 As	<		56 Ba	<	
15 P	<		34 Se	<		SumLa..Lu	0.053	0.071
16 S	0.0089	0.0008	35 Br	<		72 Hf	<2e	0.022
16 So	<		37 Rb	<		73 Ta	<	
17 Cl	0.017	0.001	38 Sr	<		74 W	<	
18 Ar	<		39 Y	<		75 Re	<	
19 K	<2e	0.0007	40 Zr	<		76 Os	<	
20 Ca	0.0024	0.0009	41 Nb	<		77 Ir	0.040	0.008
21 Sc	<2e	0.001	42 Mo	0.005	0.002	78 Pt	<	
22 Ti	0.003	0.001	44 Ru	<		79 Au	<2e	0.006
23 V	<		45 Rh	<		80 Hg	<2e	0.006
24 Cr	<		46 Pd	<		81 Tl	<	
25 Mn	<		47 Ag	<		82 Pb	<	
26 Fe	<		48 Cd	<		83 Bi	<	
27 Co	<		49 In	<		90 Th	0.009	0.003
28 Ni	<		50 Sn	<		92 U	<	

==== Light Elements =====		==== Noble Elements =====		===== Lanthanides =======	
4 Be		44 Ru	<	57 La	0.030	0.006
5 B		45 Rh	<	58 Ce	<
6 C		46 Pd	<	59 Pr	<
7 N		47 Ag	<	60 Nd	<
8 O		75 Re	<	62 Sm	<
9 F	<	76 Os	<	63 Eu	<2e	0.002
		77 Ir	0.040	0.008	64 Gd	<2e	0.002
		78 Pt	<	65 Tb	<2e	0.002
		79 Au	<2e	0.006	66 Dy	<
				67 Ho	<2e	0.015
				68 Er	0.014	0.004
				69 Tm	<2e	0.004
				70 Yb	<
				71 Lu	<2e	0.014

```
KnownConc= 0                   REST= 0                    D/S= 0
Sum Conc's before normalisation to 100% :    99.5 %
```

XRF Analyses of Manufactured Ingots

Atom No. Element	14-00-01 Cu		14-00-03 Cu		14-00-04 Cu		15-00-001 Cu		14-01-01 Cu		14-01-04 Ni		15-01-01 Si		15-01-02 Fe		14-01-05 Co		14-01-06 Au/Ag		15-01-04 W/Ta/Hf		15-01-03 V/Cr/Mn/Fe		14-01-07 Pb/Zn/Sn		14-01-08 Sn/Na/K/Mg	
Major Elements	Axial	Radial	Axial	Radial	Axial	Radial	Axial	Radial	Axial	Radial	Axial	Radial	Axial	Radial	Axial	Radial	Axial	Radial	Axial	Radial	Axial	Radial	Axial	Radial	Axial	Radial	Axial	Radial
11 Na																												
12 Mg																												
13 Al																												
14 Si																												
19 K																												
23 V																												
24 Cr																												
25 Mn																												
26 Fe																												
27 Co																												
28 Ni																												
29 Cu																												
30 Zn																												
47 Ag																												
50 Sn																												
72 Hf																												
74 W																												
79 Au																												
82 Pb																												
Minor Elements																												
4-9 Be-F																												
15 P																												
16+ S																												
17 Cl																												
18 Ar																												
20 Ca																												
21 Sc																												
22 Ti																												
31 Ga																												
32 Ge																												
40 Zr																												
41 Nb																												
42 Mo																												
44 Ru																												
45 Rh																												
48 Cd																												
51 Sb																												
56 Ba																												
57-71 Lanthanide																												
75 Re																												
77 Ir																												
90 Th																												
Sum of content Before normaliz.	100	99.6			99.5	85.1		94.5	101.7	102.1		100.5	63.4	73.7	99.5	97.3	102	101.3		103	101	101.9	105.1	93.9	111.4	115.6	94.5	95.1

Table 4

Table 5

```
Quantum Catalytics, LLC          ANALYSIS REPORT              by Uniquant
------------------------------------------------------------------------
OLD.347 of 19-Jun-01                                   Today 19-Jun-01
Spectrometers configuration:  ARL 8410 Rh 60kV LiF220 LiF420 Ge111 TlAP
Sample ident  =  14-01-01    AXIAL
Further info  =  14-01-01 AXIAL 6/19
Kappa list    =  15-Nov-94    Channel list =  18-Jun-01
Calculated as :  Elements    Spectral impurity data : CAL.909 Teflon (7/94)
X-ray path    =  Vacuum       Film type    = No supporting film
Case number   =  0   Known Area, %Rest, Diluent/Sample and Mass/Area
Eff.Diam.     = 25.00 mm      Eff.Area     = 490.6 mm2
KnownConc     =  0    %
Rest          =  0    %
Dil/Sample    =  0
Viewed Mass   = 18000.00 mg
Sample Height =  5   mm
              <    means that the concentration is <  20 ppm
              <2e  means wt% < 2 StdErr. The + in Z+El means involved in Sum=100%
```

Z	wt%	StdErr	Z	wt%	StdErr	Z	wt%	StdErr
SumBe..F	0	0.034	29+Cu	98.07	0.07	51 Sb	<	
11+Na	0.046	0.009	30+Zn	<		52 Te	<	
12 Mg	<		31+Ga	0.006	0.003	53 I	<	
13+Al	0.93	0.04	32 Ge	<		55 Cs	<	
14+Si	0.89	0.04	33 As	<		56 Ba	<2e	0.003
			34 Se	<		SumLa..Lu	0.003	0.058
15+P	<		35 Br	<		72+Hf	<	
16 S			37 Rb	<		73+Ta	<	
16+So	0.0087	0.0008	38 Sr	<		74 W	<	
17+Cl	0.0107	0.0010	39 Y	<		75 Re	<	
18+Ar	0.009	0.002	40 Zr	<		76 Os	<	
19 K	<		41 Nb	<		77+Ir	0.025	0.008
20+Ca	0.0078	0.0008	42 Mo	<		78 Pt	<2e	0.007
21 Sc	<		44 Ru	<		79 Au	<	
22 Ti	<		45 Rh	<2e	0.002	80 Hg	<2e	0.006
23 V	<		46 Pd	<		81 Tl	<	
24 Cr	<		47 Ag	<2e	0.002	82 Pb	<2e	0.003
25 Mn	.		48 Cd	<		83 Bi	<	
26 Fe	<		49 In	<		90 Th	<2e	0.003
27 Co	<		50 Sn	<		92 U	<	
28 Ni	<							

```
==== Light Elements =====   ==== Noble Elements =====   ===== Lanthanides =======
   4 Be                        44 Ru    <                 57 La    <
   5 B                         45 Rh    <2e    0.002      58 Ce    <
   6 C                         46 Pd    <                 59 Pr    <
   7 N                         47 Ag    <2e    0.002      60 Nd    <
   8 O                         75 Re    <                 62 Sm    <
   9 F       <                 76 Os    <                 63 Eu    <2e    0.001
                               77+Ir    0.025  0.008      64 Gd    <
                               78 Pt    <2e    0.007      65 Tb    <
                               79 Au    <                 66 Dy    <
                                                          67 Ho    <
                                                          68 Er    <
                                                          69 Tm    <
                                                          70 Yb    <
                                                          71+Lu    <

KnownConc= 0                REST= 0                  D/S= 0
Sum Conc's before normalisation to 100% :   101.7 %
```

Table 6

```
Quantum Catalytics, LLC              ANALYSIS REPORT                 by Uniquant
-------------------------------------------------------------------------------
OLD.346 of 19-Jun-01                                          Today 19-Jun-01
Spectrometers configuration:  ARL 8410 Rh 60kV LiF220 LiF420 Ge111 TlAP
Sample ident   =  14-01-01      RADIAL
Further info   =  14-01-01 RADIAL 6/19
Kappa list     =  15-Nov-94      Channel list =  18-Jun-01
Calculated as :  Elements        Spectral impurity data : CAL.909 Teflon (7/94)
X-ray path     =  Vacuum         Film type  = No supporting film
Case number    =  0   Known Area, %Rest, Diluent/Sample and Mass/Area
Eff.Diam.      = 25.00 mm        Eff.Area     = 490.6 mm2
KnownConc      =  0   %
Rest           =  0   %
Dil/Sample     =  0
Viewed Mass    = 18000.00 mg
Sample Height  =  5  mm
               <    means that the concentration is <  20 ppm
               <2e  means wt% < 2 StdErr. The + in Z+El means involved in Sum=100%
```

z	wt%	StdErr	z	wt%	StdErr	z	wt%	StdErr
SumBe..F	0.071	0.036	29+Cu	98.69	0.06	51 Sb	<	
11+Na	0.068	0.009	30+Zn	<		52 Te	<	
12 Mg	<		31+Ga	0.006	0.003	53 I	<	
13+Al	0.67	0.03	32 Ge	<		55 Cs	<	
14+Si	0.51	0.03	33 As	<		56 Ba	<	
15+P	<		34 Se	<		SumLa..Lu	0.014	0.062
16 S			35 Br	<		72+Hf	<	
16+So	0.0026	0.0004	37 Rb	<		73+Ta	<	
17+Cl	0.0065	0.0009	38 Sr	<		74 W	<	
18+Ar	0.006	0.001	39 Y	<		75 Re	<	
19 K	<		40 Zr	<		76 Os	<	
20 Ca	<		41 Nb	<		77+Ir	<2e	0.008
21+Sc	0.0040	0.0007	42+Mo	0.005	0.002	78 Pt	<	
22 Ti	<		44 Ru	<2e	0.002	79 Au	<2e	0.007
23 V	<		45 Rh	<		80 Hg	<	
24+Cr	<		46 Pd	<		81 Tl	<	
25 Mn	<		47+Ag	0.004	0.002	82 Pb	<	
26 Fe	<		48 Cd	<		83 Bi	<	
27+Co	<		49 In	<		90 Th	<2e	0.003
28 Ni	<		50 Sn	<		92 U	<	

==== Light Elements =====			==== Noble Elements =====			===== Lanthanides ======		
4 Be			44 Ru	<2e	0.002	57 La	<	
5 B			45 Rh	<		58 Ce	<	
6 C			46 Pd	<		59 Pr	<	
7 N			47+Ag	0.004	0.002	60 Nd	<	
8 O			75 Re	<		62 Sm	<	
9 F	<2e	0.036	76 Os	<		63 Eu	<	
			77+Ir	<2e	0.008	64 Gd	<	
			78 Pt	<		65 Tb	<	
			79 Au	<2e	0.007	66 Dy	<	
						67 Ho	<	
						68+Er	0.012	0.003
						69 Tm	<	
						70 Yb	<	
						71+Lu	<	

```
KnownConc= 0                    REST= 0                 D/S= 0
Sum Conc's before normalisation to 100% :   102.1 %
```

Table 7

```
Quantum Catalytics, LLC          ANALYSIS REPORT              by Uniquant
-------------------------------------------------------------------------
OLD.236 of 23-Apr-01                                    Today 23-Apr-01
Spectrometers configuration: ARL 8410 Rh 60kV LiF220 LiF420 Ge111 TlAP
Sample ident  =  14-01-01 Cu  SECTION TOP
Further info  =  14-01-01 MIDDLE SECTION TOP FACE AXIAL
Kappa list    =  15-Nov-94    Channel list = 19-Apr-01
Calculated as :  Elements     Spectral impurity data : CAL.909 Teflon (7/94)
X-ray path    =  Vacuum       Film type  = No supporting film
Case number   =  0   Known Area, %Rest, Diluent/Sample and Mass/Area
Eff.Diam.     =  25.00 mm     Eff.Area     = 490.6 mm2
KnownConc     =  0     %
Rest          =  0     %
Dil/Sample    =  0
Viewed Mass   =  18000.00 mg
Sample Height =  5   mm
          <     means that the concentration is <  20 ppm
          <2e   means wt% < 2 StdErr. The + in Z+El means involved in Sum=100%
```

Z	wt%	StdErr	Z	wt%	StdErr	Z	wt%	StdErr
SumBe..F	0	0.037	29+Cu	99.71	0.03	51 Sb	<	
11+Na	0.098	0.011	30+Zn	<		52 Te	<	
12 Mg	<2e	0.004	31 Ga	<2e	0.003	53 I	<	
13+Al	0.066	0.005	32 Ge	<		55+Cs	0.007	0.003
14 Si	<		33 As	<		56 Ba	<	
15+P	<		34 Se	<		SumLa..Lu	0.024	0.074
16+S	0.014	0.001	35 Br	<		72+Hf	<	
16 So			37 Rb	<		73+Ta	<	
17+Cl	0.028	0.002	38 Sr	<		74 W	<	
18+Ar	0.019	0.002	39 Y	<		75 Re	<	
19+K	0.0021	0.0007	40 Zr	<		76 Os	<	
20+Ca	<		41 Nb	<		77+Ir	0.024	0.008
21 Sc	<		42 Mo	<		78 Pt	<	
22+Ti	0.0021	0.0009	44 Ru	<		79 Au	<	
23 V	<		45 Rh	<		80 Hg	<	
24 Cr	<		46 Pd	<		81 Tl	<	
25 Mn	<		47 Ag	<		82 Pb	<	
26 Fe	<		48+Cd	0.004	0.002	83 Bi	<	
27 Co	<		49 In	<		90+Th	0.007	0.003
28 Ni	<		50 Sn	<		92 U	<	

==== Light Elements =====			==== Noble Elements =====			===== Lanthanides =======		
4 Be		-	44 Ru	<		57+La	0.015	0.004
5 B			45 Rh	<		58 Ce	<	
6 C			46 Pd	<		59 Pr	<	
7 N			47 Ag	<		60 Nd	<	
8 O			75 Re	<		62 Sm	<	
9 F	<		76 Os	<		63 Eu	<	
			77+Ir	0.024	0.008	64 Gd	<2e	0.002
			78 Pt	<		65 Tb	<	
			79 Au	<		66 Dy	<	
						67 Ho	<	
						68 Er	<2e	0.004
						69 Tm	<2e	0.005
						70 Yb	<	
						71+Lu	<	

```
   KnownConc= 0                  REST= 0                    D/S= 0
   Sum Conc's before normalisation to 100% :  102.7 %
```

Table 8

```
Quantum Catalytics, LLC          ANALYSIS REPORT                   by Uniquant
-----------------------------------------------------------------------------
OLD.244 of 19-Apr-01                                   Today 24-Apr-01
Spectrometers configuration: ARL 8410 Rh 60kV LiF220 LiF420 Ge111 TlAP
Sample ident    =  14-01-01 MID BOTTOM FACE
Further info    =  14-01-01 MIDDLE SECTION BOTTOM FCE AXIAL
Kappa list      =  15-Nov-94    Channel list =  19-Apr-01
Calculated as   :  Elements     Spectral impurity data : CAL.909 Teflon (7/94)
X-ray path      =  Vacuum       Film type    = No supporting film
Case number     =  0    Known Area, %Rest, Diluent/Sample and Mass/Area
Eff.Diam.       = 25.00 mm      Eff.Area     = 490.6 mm2
KnownConc       =  0    %
Rest            =  0    %
Dil/Sample      =  0
Viewed Mass     = 18000.00 mg
Sample Height   =  5   mm
            <   means that the concentration is < 20 ppm
           <2e  means wt% < 2 StdErr. The + in Z+El means involved in Sum=100%
```

Z	wt%	StdErr	Z	wt%	StdErr	Z	wt%	StdErr
SumBe..F	0	0.037	29+Cu	98.42	0.06	51 Sb	<	
11+Na	0.21	0.01	30+Zn	<		52 Te	<	
12+Mg	0.013	0.004	31+Ga	0.006	0.003	53 I	<	
13+Al	0.80	0.04	32 Ge	<		55 Cs	<	
14+Si	0.34	0.02	33 As	<		56 Ba	<2e	0.003
15+P	<		34 Se	<		SumLa..Lu	0.018	0.071
16+S	0.031	0.003	35 Br	<		72+Hf	<2e	0.022
16 So			37 Rb	<		73+Ta	<	
17+Cl	0.070	0.006	38+Sr	0.0024	0.0010	74 W	<	
18+Ar	0.019	0.002	39 Y	<		75 Re	<	
19+K	0.020	0.002	40 Zr	<		76 Os	<2e	0.006
20+Ca	0.012	0.001	41 Nb	<		77+Ir	0.033	0.008
21 Sc	<		42+Mo	<2e	0.002	78 Pt	<	
22+Ti	0.003	0.001	44 Ru	<		79 Au	<2e	0.006
23 V	<		45 Rh	<2e	0.002	80 Hg	<2e	0.006
24 Cr	<		46 Pd	<		81 Tl	<	
25 Mn	<		47 Ag	<		82+Pb	0.009	0.003
26 Fe	<2e	0.001	48+Cd	0.004	0.002	83 Bi	<	
27+Co	<		49 In	<		90 Th	<	
28 Ni	<		50 Sn	<		92 U	<	

==== Light Elements =====			==== Noble Elements ====			===== Lanthanides ======		
4 Be			44 Ru	<		57 La	<2e	0.005
5 B			45 Rh	<2e	0.002	58 Ce	<	
6 C			46 Pd	<		59 Pr	<	
7 N			47 Ag	<		60 Nd	<	
8 O			75 Re	<		62 Sm	<	
9 F	<		76 Os	<2e	0.006	63 Eu	<2e	0.002
			77+Ir	0.033	0.008	64 Gd	<	
			78 Pt	<		65 Tb	<	
			79 Au	<2e	0.006	66 Dy	<	
						67 Ho	<	
						68+Er	0.013	0.004
						69 Tm	<	
						70 Yb	<	
						71+Lu	<	

```
KnownConc= 0                    REST= 0                 D/S= 0
Sum Conc's before normalisation to 100% :  103.1 %
```

Table 9

```
Quantum Catalytics, LLC          ANALYSIS REPORT                    by Uniquant
------------------------------------------------------------------------------
OLD.252 of 19-Apr-01                                       Today 25-Apr-01
Spectrometers configuration: ARL 8410 Rh 60kV LiF220 LiF420 Ge111 TlAP
Sample ident  =  14-01-01 MID SECT TOP FAC
Further info  =  14-01-01 MIDDLE SECTION TOP FACE 4/25/01
Kappa list    =  15-Nov-94     Channel list = 19-Apr-01
Calculated as :  Elements      Spectral impurity data : CAL.909 Teflon (7/94)
X-ray path    =  Vacuum        Film type    = No supporting film
Case number   =  0     Known Area, %Rest, Diluent/Sample and Mass/Area
Eff.Diam.     = 25.00 mm        Eff.Area    = 490.6 mm2
KnownConc     =  0    %
Rest          =  0    %
Dil/Sample    =  0
Viewed Mass   = 18000.00 mg
Sample Height =  5  mm
```

```
         <     means that the concentration is <  20 ppm
         <2e   means wt% < 2 StdErr. The + in Z+El means involved in Sum=100%
```

Z	wt%	StdErr	Z	wt%	StdErr	Z	wt%	StdErr
SumBe..F	0	0.038	29+Cu	99.66	0.03	51 Sb	<	
11+Na	0.034	0.011	30+Zn	<		52 Te	<	
12 Mg	<		31 Ga	<2e	0.003	53 I	<	
13+Al	0.15	0.01	32 Ge	<		55 Cs	<	
14+Si	0.033	0.005	33 As	<2e	0.004	56 Ba	<2e	0.003
15+P	<		34 Se	<		SumLa..Lu	0.026	0.075
16+S	0.017	0.002	35 Br	<		72+Hf	<	
16 So			37 Rb	<		73+Ta	<	
17+Cl	0.027	0.002	38+Sr	0.003	0.001	74 W	<	
18+Ar	0.017	0.002	39 Y	<		75 Re	<	
19+K	0.0021	0.0007	40 Zr	<		76 Os	<2e	0.007
20+Ca	0.0056	0.0007	41 Nb	<		77+Ir	0.026	0.008
21 Sc	<		42 Mo	<		78 Pt	<	
22+Ti	0.0023	0.0010	44 Ru	<		79 Au	<	
23 V	<		45 Rh	<		80 Hg	<	
24 Cr	<		46 Pd	<		81 Tl	<	
25 Mn	<		47 Ag	<		82 Pb	<	
26 Fe	<		48 Cd	<		83 Bi	<2e	0.005
27 Co	<		49 In	<		90 Th	<2e	0.003
28 Ni	<		50 Sn	<		92 U	<	

==== Light Elements =====			==== Noble Elements =====			===== Lanthanides =======		
4 Be			44 Ru	<		57+La	0.021	0.004
5 B			45 Rh	<		58 Ce	<	
6 C			46 Pd	<		59 Pr	<	
7 N			47 Ag	<		60 Nd	<	
8 O			75 Re	<		62 Sm	<	
9 F	<		76 Os	<2e	0.007	63 Eu	<	
			77+Ir	0.026	0.008	64 Gd	<	
			78 Pt	<		65 Tb	<	
			79 Au	<		66 Dy	<	
						67 Ho	<	
						68 Er	<	
						69 Tm	<2e	0.005
						70 Yb	<	
						71+Lu	<	

```
KnownConc= 0                  REST= 0              D/S= 0
Sum Conc's before normalisation to 100% :  102.6 %
```

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Table 10

```
Quantum Catalytics, LLC          ANALYSIS REPORT                by Uniquant
----------------------------------------------------------------------------
OLD.255 of 19-Apr-01                                    Today 25-Apr-01
Spectrometers configuration: ARL 8410 Rh 60kV LiF220 LiF420 Ge111 TlAP
Sample ident  =  14-01-01 MID SECT TOP FAC
Further info  =  14-01-01 MIDDLE SECTION TOP FACE 4/25/01
Kappa list    =  15-Nov-94    Channel list =  19-Apr-01
Calculated as :  Elements     Spectral impurity data : CAL.909 Teflon (7/94)
X-ray path    =  Vacuum       Film type    = No supporting film
Case number   =  0   Known Area, %Rest, Diluent/Sample and Mass/Area
Eff.Diam.     =  25.00 mm     Eff.Area     = 490.6 mm2
KnownConc     =  0     %
Rest          =  0     %
Dil/Sample    =  0
Viewed Mass   =  18000.00 mg
Sample Height =  5   mm
            <    means that the concentration is <   20 ppm
            <2e  means wt% < 2 StdErr. The + in Z+El means involved in Sum=100%
```

Z	wt%	StdErr	Z	wt%	StdErr	Z	wt%	StdErr
SumBe..F	0	0.035	29+Cu	99.70	0.03	51 Sb	<	
11+Na	<2e	0.011	30+Zn	<		52 Te	<	
12 Mg	<		31 Ga	<2e	0.003	53 I	<	
13+Al	0.14	0.01	32 Ge	<		55 Cs	<	
14+Si	0.028	0.006	33 As	<		56 Ba	<	
15+P	<		34 Se	<		SumLa..Lu	0.019	0.075
16+S	0.018	0.002	35 Br	<		72+Hf	<	
16 So			37 Rb	<		73+Ta	<	
17+Cl	0.028	0.002	38 Sr	<		74 W	<	
18+Ar	0.020	0.002	39 Y	<		75 Re	<	
19+K	0.0033	0.0007	40 Zr	<		76 Os	<	
20+Ca	0.0058	0.0007	41 Nb	<		77+Ir	0.021	0.008
21 Sc	<		42 Mo	<		78 Pt	<2e	0.008
22+Ti	0.0028	0.0009	44 Ru	<		79 Au	<	
23 V	<		45 Rh	<		80 Hg	<2e	0.006
24 Cr	<		46 Pd	<		81 Tl	<	
25 Mn	<		47 Ag	<2e	0.002	82 Pb	<	
26 Fe	<		48 Cd	<		83 Bi	<	
27 Co	<		49 In	<		90 Th	0.006	0.003
28 Ni	<		50 Sn	<		92 U	<	

==== Light Elements =====			==== Noble Elements =====			===== Lanthanides ======		
4 Be			44 Ru	<		57+La	0.016	0.003
5 B			45 Rh	<		58 Ce	<	
6 C			46 Pd	<		59 Pr	<	
7 N			47 Ag	<2e	0.002	60 Nd	<	
8 O			75 Re	<		62 Sm	<	
9 F	<		76 Os	<		63 Eu	<	
			77+Ir	0.021	0.008	64 Gd	<	
			78 Pt	<2e	0.008	65 Tb	<	
			79 Au	<		66 Dy	<	
						67 Ho	<	
						68 Er	<	
						69 Tm	<2e	0.005
						70 Yb	<	
						71+Lu	<	

```
KnownConc= 0                    REST= 0                 D/S= 0
Sum Conc's before normalisation to 100% :  102.5 %
```

Table 11

```
Quantum Catalytics, LLC        ANALYSIS REPORT                    by Uniquant
-----------------------------------------------------------------------------
OLD.259 of 19-Apr-01                                    Today 26-Apr-01
Spectrometers configuration:  ARL 8410 Rh 60kV LiF220 LiF420 Ge111 TlAP
Sample ident  = 14-01-01 BOT BOTTOM
Further info  = 14-01-01 MIDDLE SECTION BOT FACE 4/25/01
Kappa list    = 15-Nov-94    Channel list = 19-Apr-01
Calculated as : Elements     Spectral impurity data : CAL.909 Teflon (7/94)
X-ray path    = Vacuum       Film type    = No supporting film
Case number   = 0   Known Area, %Rest, Diluent/Sample and Mass/Area
Eff.Diam.     = 25.00 mm      Eff.Area     = 490.6 mm2
KnownConc     = 0    %
Rest          = 0    %
Dil/Sample    = 0
Viewed Mass   = 18000.00 mg
Sample Height = 5  mm
              <    means that the concentration is <  20 ppm
             <2e  means wt% < 2 StdErr. The + in Z+El means involved in Sum=100%
     Z       wt%    StdErr     Z        wt%    StdErr     Z        wt%    StdErr
    ======================     =======================    ========================
  SumBe..F   0      0.037    29+Cu     98.53   0.06    51 Sb      <
  11+Na      0.14   0.01     30+Zn     <               52 Te      <
  12 Mg      <2e    0.004    31 Ga     <2e     0.003   53 I       <
  13+Al      0.78   0.04     32 Ge     <               55 Cs      <
  14+Si      0.35   0.02     33 As     <               56 Ba      <

  15+P       <               34 Se     <               SumLa..Lu  0.024   0.071
  16 S                       35 Br     <               72+Hf      <
  16+So      0.032  0.003    37 Rb     <               73+Ta      <
  17+Cl      0.061  0.005    38 Sr     <               74 W       <
  18+Ar      0.018  0.002    39 Y      <               75 Re      <

  19+K       0.021  0.002    40 Zr     <               76 Os      <
  20+Ca      0.013  0.001    41 Nb     <               77+Ir      0.026   0.008
  21 Sc      <               42 Mo     <               78 Pt      <
  22 Ti      <2e    0.001    44 Ru     <               79 Au      <
  23 V       <               45 Rh     <               80 Hg      <2e     0.006

  24 Cr      <               46 Pd     <               81 Tl      <
  25+Mn      <               47 Ag     <               82+Pb      0.007   0.003
  26+Fe      0.003  0.001    48 Cd     <               83 Bi      <
  27 Co      <               49 In     <               90 Th      <2e     0.003
  28 Ni      <               50 Sn     <               92 U       <

  ==== Light Elements =====  ==== Noble Elements =====  ===== Lanthanides =======
   4 Be                      44 Ru     <               57 La      <2e     0.004
   5 B                       45 Rh     <               58 Ce      <
   6 C                       46 Pd     <               59 Pr      <
   7 N                       47 Ag     <               60 Nd      <
   8 O                       75 Re     <               62 Sm      <
   9 F       <               76 Os     <               63+Eu      0.005   0.002
                             77+Ir     0.026   0.008   64 Gd      <2e     0.002
                             78 Pt     <               65 Tb      <
                             79 Au     <               66 Dy      <
                                                       67 Ho      <
                                                       68+Er      0.012   0.004
                                                       69 Tm      <
                                                       70 Yb      <
                                                       71+Lu      <

  KnownConc= 0              REST= 0              D/S= 0
  Sum Conc's before normalisation to 100% :  102.9 %
```

Table 12

```
Quantum Catalytics, LLC          ANALYSIS REPORT                by Uniquant
--------------------------------------------------------------------------------
OLD.260 of 19-Apr-01                                      Today 26-Apr-01
Spectrometers configuration: ARL 8410 Rh 60kV LiF220 LiF420 Ge111 TlAP
Sample ident   =  14-01-01 BOT BOTTOM
Further info   =  14-01-01 MID SECT BOT FACE 4/25/01  BTB
Kappa list     =  15-Nov-94    Channel list =  19-Apr-01
Calculated as  :  Elements     Spectral impurity data : CAL.909 Teflon (7/94)
X-ray path     =  Vacuum       Film type    = No supporting film
Case number    =  0 . Known Area, %Rest, Diluent/Sample and Mass/Area
Eff.Diam.      = 25.00 mm       Eff.Area     = 490.6 mm2
KnownConc      = 0      %
Rest           = 0    %
Dil/Sample     = 0
Viewed Mass    = 18000.00 mg
Sample Height  = 5   mm
         <    means that the concentration is <  20 ppm
         <2e  means wt% < 2 StdErr. The + in Z+El means involved in Sum=100%
```

Z	wt%	StdErr	Z	wt%	StdErr	Z	wt%	StdErr
SumBe..F	0	0.036	29+Cu	98.55	0.06	51 Sb	<	
11+Na	0.12	0.01	30+Zn	<		52 Te	<	
12 Mg	<2e	0.004	31+Ga	0.010	0.003	53 I	<	
13+Al	0.77	0.04	32 Ge	<2e	0.002	55+Cs	0.006	0.003
14+Si	0.34	0.02	33 As	<2e	0.004	56 Ba	0.006	0.003
15+P	<		34 Se	<		SumLa..Lu	0.013	0.071
16+S	0.034	0.003	35 Br	<		72+Hf	<2e	0.022
16 So			37 Rb	<		73+Ta	<	
17+Cl	0.064	0.005	38 Sr	<		74 W	<	
18+Ar	0.018	0.002	39 Y	<		75 Re	<	
19+K	0.021	0.002	40 Zr	<		76 Os	<2e	0.007
20+Ca	0.014	0.001	41 Nb	<		77+Ir	0.018	0.007
21 Sc	<		42 Mo	<2e	0.002	78 Pt	<	
22 Ti	<		44 Ru	<		79+Au	0.014	0.007
23 V	<		45 Rh	<		80 Hg	<2e	0.006
24 Cr	<		46 Pd	<		81 Tl	<	
25 Mn	<		47 Ag	<2e	0.002	82 Pb	<2e	0.003
26 Fe	<2e	0.001	48 Cd	<		83+Bi	0.013	0.005
27 Co	<		49 In	<		90 Th	<2e	0.003
28 Ni	<		50 Sn	<		92 U	<	

```
==== Light Elements =====   ==== Noble Elements =====   ===== Lanthanides =======
   4 Be                        44 Ru    <                57+La    <2e    0.005
   5 B                         45 Rh    <                58 Ce    <2e    0.002
   6 C                         46 Pd    <                59 Pr    <
   7 N                         47 Ag    <2e    0.002     60 Nd    <
   8 O                         75 Re    <                62 Sm    <
   9 F        <                76 Os    <2e    0.007     63 Eu    <
                               77+Ir    0.018  0.007     64 Gd    <
                               78 Pt    <                65 Tb    <2e    0.002
                               79+Au    0.014  0.007     66 Dy    <
                                                         67 Ho    <
                                                         68 Er    <
                                                         69 Tm    <
                                                         70 Yb    <
                                                         71+Lu    <

KnownConc= 0                     REST= 0              D/S= 0
Sum Conc's before normalisation to 100% :  103.1 %
```

Table 13

```
Quantum Catalytics, LLC          ANALYSIS REPORT              by Uniquant
-------------------------------------------------------------------------
OLD.261 of 19-Apr-01                                  Today 26-Apr-01
Spectrometers configuration: ARL 8410 Rh 60kV LiF220 LiF420 Ge111 TlAP
Sample ident   = 14-01-01 BOT BOTTOM
Further info   = 14-01-01 MID SECT BOT FACE 4/25/01 3 HR
Kappa list     = 15-Nov-94   Channel list =  19-Apr-01
Calculated as :  Elements    Spectral impurity data : CAL.909 Teflon (7/94)
X-ray path     = Vacuum       Film type   = No supporting film
Case number    = 0  Known Area, %Rest, Diluent/Sample and Mass/Area
Eff.Diam.      = 25.00 mm     Eff.Area     = 490.6 mm2
KnownConc      = 0     %
Rest           = 0     %
Dil/Sample     = 0
Viewed Mass    = 18000.00 mg
Sample Height  = 5   mm
                <     means that the concentration is <  20 ppm
                <2e means wt% < 2 StdErr. The + in Z+El means involved in Sum=100%
    Z       wt%    StdErr    Z        wt%    StdErr     Z          wt%    StdErr
==========================   ==========================   ==========================
SumBe..F   0      0.038     29+Cu   98.50    0.06      51 Sb       <
11+Na      0.15   0.01      30+Zn    <                 52 Te       <
12+Mg      0.014  0.004     31+Ga    0.009   0.003     53 I        <
13+Al      0.79   0.04      32 Ge    <                 55 Cs       <
14+Si      0.35   0.02      33 As    <                 56 Ba       <

15+P       <                34 Se    <                 SumLa..Lu   0.016   0.071
16+S       0.036  0.003     35 Br    <                 72+Hf       <
16 So                       37 Rb    <                 73+Ta       <
17+Cl      0.062  0.005     38 Sr    <                 74 W        <
18+Ar      0.018  0.002     39 Y     <                 75 Re       <

19+K       0.020  0.002     40 Zr    <                 76 Os       <
20+Ca      0.0108 0.0010    41 Nb    <                 77+Ir       0.016   0.007
21+Sc      0.0020 0.0009    42 Mo    <                 78 Pt       <2e     0.007
22+Ti      <2e    0.001     44 Ru    <                 79 Au       <
23 V       <                45 Rh    <                 80 Hg       <2e     0.006

24 Cr      <                46 Pd    <                 81 Tl       <
25 Mn      <                47 Ag    <2e     0.002     82 Pb       0.006   0.003
26 Fe      <2e    0.001     48+Cd    0.004   0.002     83 Bi       <
27+Co      <                49 In    <                 90 Th       <2e     0.003
28 Ni      <                50 Sn    <                 92 U        <

==== Light Elements =====   ==== Noble Elements =====   ===== Lanthanides =======
  4 Be                       44 Ru    <                 57 La       <2e     0.004
  5 B                        45 Rh    <                 58 Ce       <
  6 C                        46 Pd    <                 59 Pr       <
  7 N                        47 Ag    <2e     0.002     60 Nd       <
  8 O                        75 Re    <                 62 Sm       <
  9 F       <                76 Os    <                 63 Eu       <
                             77+Ir    0.016   0.007     64 Gd       <
                             78 Pt    <2e     0.007     65 Tb       <
                             79 Au    <                 66 Dy       <
                                                        67 Ho       <
                                                        68+Er       0.013   0.004
                                                        69 Tm       <
                                                        70 Yb       <
                                                        71+Lu       <

KnownConc= 0                  REST= 0               D/S= 0
Sum Conc's before normalisation to 100% :  102.9 %
```

Table 14

```
Quantum Catalytics, LLC          ANALYSIS REPORT              by Uniquant
------------------------------------------------------------------------------
OLD.320 of 24-May-01                                   Today 25-May-01
Spectrometers configuration: ARL 8410 Rh 60kV LiF220 LiF420 Ge111 TlAP
Sample ident  =  14-01-04 Ni AXIAL BLK
Further info  =  14-01-04 Ni AXIAL BLK 5/25
Kappa list    =  15-Nov-94    Channel list =  24-May-01
Calculated as :  Elements     Spectral impurity data : CAL.909 Teflon (7/94)
X-ray path    =  Vacuum       Film type    = No supporting film
Case number   =  0    Known Area, %Rest, Diluent/Sample and Mass/Area
Eff.Diam.     =  25.00 mm     Eff.Area     = 490.6 mm2
KnownConc     =  0    %
Rest          =  0    %
Dil/Sample    =  0
Viewed Mass   =  18000.00 mg
Sample Height =  5  mm
         <     means that the concentration is <  20 ppm
         <2e   means wt% < 2 StdErr. The + in Z+El means involved in Sum=100%
```

Z	wt%	StdErr	Z	wt%	StdErr	Z	wt%	StdErr
SumBe..F	0	0.035	29 Cu	<		51 Sb	<	
11+Na	0.031	0.009	30 Zn	<		52 Te	<	
12 Mg	<		31 Ga	<		53 I	<	
13+Al	2.11	0.07	32 Ge	<		55 Cs	<	
14+Si	0.030	0.005	33 As	<		56 Ba	<2e	0.003
15+P	0.0020	0.0003	34 Se	<		SumLa..Lu	0.69	1.22
16 S			35 Br	<		72 Hf	<2e	0.007
16+So	0.0042	0.0004	37 Rb	<		73+Ta	<	
17+Cl	<		38 Sr	<		74+W	<	
18 Ar	<		39 Y	<		75 Re	<	
19 K	<		40+Zr	0.027	0.002	76 Os	<	
20 Ca	<		41 Nb	<		77 Ir	<	
21+Sc	0.0024	0.0008	42 Mo	<2e	0.002	78 Pt	<	
22 Ti	<		44 Ru	<2e	0.002	79 Au	<	
23 V	<		45+Rh	0.005	0.002	80 Hg	<	
24 Cr	<		46 Pd	<		81 Tl	<	
25 Mn	<		47 Ag	<		82 Pb	<	
26 Fe	<		48 Cd	<		83 Bi	<	
27 Co	<		49 In	<		90 Th	<	
28+Ni	97.10	0.08	50 Sn	<		92 U	<	

==== Light Elements =====			==== Noble Elements =====			===== Lanthanides =======		
4 Be			44 Ru	<2e	0.002	57 La	<	
5 B			45+Rh	0.005	0.002	58 Ce	<	
6 C			46 Pd	<		59 Pr	<	
7 N			47 Ag	<		60 Nd	<	
8 O			75 Re	<		62 Sm	<	
9 F	<		76 Os	<		63 Eu	<	
			77 Ir	<		64 Gd	<	
			78 Pt	<		65 Tb	<	
			79 Au	<		66+Dy	<	
						67+Ho	<	
						68 Er	<	
						69 Tm	<	
						70+Yb	0.69	0.27
						71 Lu	<	

```
KnownConc= 0                 REST= 0              D/S= 0
Sum Conc's before normalisation to 100% :  101.4 %
```

Table 15

```
Quantum Catalytics, LLC          ANALYSIS REPORT                    by Uniquant
-------------------------------------------------------------------------------
OLD.321 of 24-May-01                                          Today 25-May-01
Spectrometers configuration:  ARL 8410 Rh 60kV LiF220 LiF420 Ge111 T1AP
Sample ident   =  14-01-04 Ni  RADIAL BLK
Further info   =  14-01-04 Ni RADIAL BLK 5/25
Kappa list     =  15-Nov-94    Channel list = 24-May-01
Calculated as :  Elements      Spectral impurity data : CAL.909 Teflon (7/94)
X-ray path     =  Vacuum        Film type     = No supporting film
Case number    =  0   Known Area, %Rest, Diluent/Sample and Mass/Area
Eff.Diam.      = 25.00 mm       Eff.Area      = 490.6 mm2
KnownConc      =  0     %
Rest           =  0     %
Dil/Sample     =  0
Viewed Mass    = 18000.00 mg
Sample Height  =  5    mm
              <     means that the concentration is <  20 ppm
              <2e   means wt% < 2 StdErr. The + in Z+El means involved in Sum=100%
```

z	wt%	StdErr	z	wt%	StdErr	z	wt%	StdErr
SumBe..F	0	0.037	29 Cu	<		51 Sb	<	
11+Na	0.12	0.01	30 Zn	<		52 Te	<	
12 Mg	<		31 Ga	<		53 I	<	
13+Al	2.57	0.07	32+Ge	0.005	0.002	55 Cs	<2e	0.002
14+Si	0.14	0.01	33 As	<		56 Ba	0.006	0.003
15+P	<		34 Se	<		SumLa..Lu	0.65	1.21
16 S			35 Br	<		72 Hf	<2e	0.007
16+So	0.026	0.002	37 Rb	<		73+Ta	<	
17+Cl	0.0050	0.0007	38 Sr	<		74+W	<	
18+Ar	0.010	0.001	39 Y	<		75 Re	<2e	0.006
19+K	0.0048	0.0007	40+Zr	0.032	0.003	76 Os	<	
20+Ca	0.011	0.001	41 Nb	<		77 Ir	<2e	0.006
21 Sc	<		42 Mo	<2e	0.002	78 Pt	<	
22 Ti	<		44 Ru	<		79 Au	<	
23 V	<		45 Rh	<		80 Hg	<2e	0.004
24 Cr	<		46 Pd	<		81 Tl	<	
25 Mn	<		47 Ag	<2e	0.002	82 Pb	<	
26 Fe	<		48 Cd	<2e	0.002	83 Bi	<	
27 Co	<		49 In	<		90 Th	<2e	0.002
28+Ni	96.43	0.09	50 Sn	<		92 U	<2e	0.002

==== Light Elements =====			==== Noble Elements =====			===== Lanthanides =======		
4 Be			44 Ru	<		57 La	<	
5 B			45 Rh	<		58 Ce	<	
6 C			46 Pd	<		59 Pr	<	
7 N			47 Ag	<2e	0.002	60 Nd	<	
8 O			75 Re	<2e	0.006	62 Sm	<	
9 F	<		76 Os	<		63 Eu	<	
			77 Ir	<2e	0.006	64 Gd	<	
			78 Pt	<		65 Tb	<2e	0.003
			79 Au	<		66+Dy	<	
						67+Ho	<	
						68 Er	<	
						69 Tm	<	
						70+Yb	0.64	0.27
						71 Lu	<	

```
KnownConc= 0                      REST= 0                  D/S= 0
Sum Conc's before normalisation to 100% :   100.5 %
```

Table 16

```
Quantum Catalytics, LLC          ANALYSIS REPORT              by Uniquant
-------------------------------------------------------------------------
OLD.327 of  1-Jun-01                                 Today  4-Jun-01
Spectrometers configuration: ARL 8410 Rh 60kV LiF220 LiF420 Ge111 T1AP
Sample ident  =  14-01-05 Co  AXIAL TOP
Further info  =  14-01-05 CO AXIAL TOP
Kappa list    =  15-Nov-94     Channel list =  24-May-01
Calculated as :  Elements      Spectral impurity data : CAL.909 Teflon (7/94)
X-ray path    =  Vacuum        Film type     = No supporting film
Case number   =  0   Known Area, %Rest, Diluent/Sample and Mass/Area
Eff.Diam.     = 25.00 mm        Eff.Area      = 490.6 mm2
KnownConc     =  0    %
Rest          =  0    %
Dil/Sample    =  0
Viewed Mass   = 18000.00 mg
Sample Height =  5   mm
              <      means that the concentration is <  20 ppm
              <2e  means wt% < 2 StdErr. The + in Z+El means involved in Sum=100%
```

Z	wt%	StdErr	Z	wt%	StdErr	Z	wt%	StdErr
SumBe..F	0	0.88	29 Cu	<		51 Sb	<	
11+Na	0.026	0.008	30 Zn	<		52 Te	<	
12 Mg	<		31 Ga	<2e	0.002	53 I	<	
13+Al	4.9	0.1	32 Ge	<		55 Cs	<	
14+Si	0.073	0.006	33 As	<		56+Ba	0.006	0.003
15+P	0.0040	0.0004	34 Se	<		SumLa..Lu	0.02	10.08
16 S			35 Br	<		72+Hf	0.014	0.006
16+So	<		37 Rb	<		73 Ta	<	
17+Cl	0.0047	0.0006	38 Sr	<		74 W	<	
18+Ar	0.011	0.001	39 Y	<		75 Re	<	
19 K	<		40+Zr	0.019	0.002	76 Os	<	
20+Ca	0.0075	0.0007	41 Nb	<		77 Ir	<2e	0.005
21+Sc	0.0022	0.0006	42 Mo	<		78 Pt	<	
22 Ti	<		44 Ru	<		79 Au	<	
23 V	<		45 Rh	<		80 Hg	<2e	0.004
24 Cr	<		46 Pd	<		81 Tl	<	
25+Mn	0.0091	0.0008	47 Ag	<		82 Pb	<2e	0.002
26+Fe	0.13	0.01	48 Cd	0.003	0.002	83 Bi	<	
27+Co	94.6	0.1	49 In	<		90 Th	<2e	0.002
28+Ni	0.16	0.01	50 Sn	<		92 U	<	

```
==== Light Elements =====    ==== Noble Elements ====    ===== Lanthanides =======
  4  Be                       44 Ru    <                  57 La    <
  5  B                        45 Rh    <                  58 Ce    <
  6  C                        46 Pd    <                  59 Pr    <
  7  N                        47 Ag    <                  60 Nd    <
  8  O                        75 Re    <                  62 Sm    <
  9+F       <                 76 Os    <                  63 Eu    <2e    0.002
                              77 Ir    <2e    0.005       64+Gd    0.016  0.003
                              78 Pt    <                  65 Tb    <
                              79 Au    <                  66 Dy    <
                                                          67+Ho    <
                                                          68+Er    <
                                                          69 Tm    <
                                                          70+Yb    <
                                                          71+Lu    <

KnownConc= 0                    REST= 0                    D/S= 0
Sum Conc's before normalisation to 100% :  102.0 %
```

Table 17

```
Quantum Catalytics, LLC          ANALYSIS REPORT                by Uniquant
--------------------------------------------------------------------------------
OLD.328 of  1-Jun-01                                     Today  4-Jun-01
Spectrometers configuration:  ARL 8410 Rh 60kV LiF220 LiF420 Ge111 TlAP
Sample ident   = 14-01-05 Co  RADIAL
Further info   = 14-01-05 CO RADIAL
Kappa list     = 15-Nov-94    Channel list =  24-May-01
Calculated as  :  Elements    Spectral impurity data : CAL.909 Teflon (7/94)
X-ray path     = Vacuum       Film type    = No supporting film
Case number    = 0   Known Area, %Rest, Diluent/Sample and Mass/Area
Eff.Diam.      = 25.00 mm     Eff.Area     = 490.6 mm2
KnownConc      = 0      %
Rest           = 0      %
Dil/Sample     = 0
Viewed Mass    = 18000.00 mg
Sample Height  = 5    mm
               <     means that the concentration is <  20 ppm
               <2e   means wt% < 2 StdErr. The + in Z+El means involved in Sum=100%
     Z       wt%      StdErr     Z       wt%     StdErr     Z         wt%      StdErr
    ==========================  ==========================  ==============================
    SumBe..F   0      0.88    29 Cu    <                 51 Sb     <
    11+Na    0.043   0.009    30 Zn    <                 52 Te     <
    12 Mg    <                31 Ga    <                 53 I      <
    13+Al    3.71    0.09     32 Ge    <                 55 Cs     <
    14+Si    0.18    0.01     33 As    <                 56 Ba     <

    15+P     0.0037  0.0004   34 Se    <                 SumLa..Lu 0.02     10.20
    16 S                      35 Br    <                 72 Hf     <2e      0.006
    16+So    <                37 Rb    <                 73 Ta     <
    17+Cl    0.0034  0.0007   38 Sr    <                 74 W      <
    18+Ar    0.010   0.001    39 Y     <                 75 Re     <2e      0.005

    19 K     <                40+Zr    0.017   0.001     76 Os     <
    20+Ca    0.0087  0.0008   41 Nb    <                 77 Ir     <2e      0.005
    21+Sc    0.0033  0.0006   42 Mo    <                 78 Pt     <
    22 Ti    <                44 Ru    <2e     0.002     79 Au     <
    23 V     <                45 Rh    <2e     0.002     80 Hg     <2e      0.004

    24+Cr    <                46 Pd    <                 81 Tl     <
    25+Mn    0.0081  0.0007   47 Ag    <                 82 Pb     <
    26+Fe    0.13    0.01     48 Cd    <                 83 Bi     <
    27+Co    95.7    0.1      49 In    <                 90 Th     <2e      0.002
    28+Ni    0.18    0.01     50 Sn    <                 92 U      <

    ==== Light Elements =====  ==== Noble Elements =====  ===== Lanthanides =======
    4 Be                      44 Ru    <2e     0.002     57 La     <
    5 B                       45 Rh    <2e     0.002     58 Ce     <
    6 C                       46 Pd    <                 59 Pr     <
    7 N                       47 Ag    <                 60 Nd     <
    8 O                       75 Re    <2e     0.005     62 Sm     <
    9+F      <                76 Os    <                 63+Eu     0.008    0.002
                             77 Ir    <2e     0.005     64+Gd     0.011    0.003
                             78 Pt    <                 65 Tb     <
                             79 Au    <                 66 Dy     <
                                                        67+Ho     <
                                                        68+Er     <
                                                        69 Tm     <
                                                        70+Yb     <
                                                        71+Lu     <

    KnownConc= 0                 REST= 0                    D/S= 0
    Sum Conc's before normalisation to 100% :   101.3 %
```

Table 18

```
Quantum Catalytics, LLC          ANALYSIS REPORT              by Uniquant
-----------------------------------------------------------------------------
OLD.330 of  4-Jun-01                                    Today  4-Jun-01
Spectrometers configuration:  ARL 8410 Rh 60kV LiF220 LiF420 Ge111 TlAP
Sample ident  =  14-01-06 Cu/ Ag/Au AX TOP
Further info  =  14-01-06 Cu/Ag/Au AXIAL TOP 6/4/01
Kappa list    =  15-Nov-94    Channel list =   4-Jun-01
Calculated as :  Elements     Spectral impurity data : CAL.909 Teflon (7/94)
X-ray path    =  Vacuum       Film type     = No supporting film
Case number   =  0   Known Area, %Rest, Diluent/Sample and Mass/Area
Eff.Diam.     =  25.00 mm      Eff.Area      = 490.6 mm2
KnownConc     =  0    %
Rest          =  0    %
Dil/Sample    =  0
Viewed Mass   =  18000.00 mg
Sample Height =  5   mm
            <     means that the concentration is <  20 ppm
            <2e   means wt% < 2 StdErr. The + in Z+El means involved in Sum=100%
```

Z	wt%	StdErr	Z	wt%	StdErr	Z	wt%	StdErr
SumBe..F	0	0.041	29+Cu	96.97	0.09	51 Sb	<	
11+Na	0.056	0.010	30+Zn	<		52 Te	<	
12 Mg	<		31+Ga	0.007	0.003	53 I	<	
13+Al	0.013	0.003	32 Ge	<		55 Cs	<	
14 Si	<		33 As	<		56 Ba	<	
15+P	<		34+Se	<		SumLa..Lu	0.012	0.071
16+S	0.0032	0.0004	35+Br	<		72+Hf	<	
16 So			37+Rb	<		73+Ta	<	
17+Cl	0.020	0.002	38 Sr	<		74 W	<	
18+Ar	0.011	0.002	39 Y	<		75+Re	0.021	0.008
19 K	<		40+Zr	0.0074	0.0010	76 Os	<	
20+Ca	0.0029	0.0008	41 Nb	<		77+Ir	0.026	0.008
21+Sc	<		42 Mo	<2e	0.002	78 Pt	<	
22 Ti	<		44 Ru	<		79+Au	1.37	0.05
23 V	<		45 Rh	<		80 Hg	<2e	0.006
24 Cr	<		46 Pd	<		81 Tl	<	
25 Mn	<		47+Ag	1.49	0.06	82 Pb	<	
26 Fe	<		48 Cd	<		83 Bi	<	
27 Co	<		49 In	<		90 Th	<2e	0.003
28 Ni	<		50+Sn	<		92 U	<	

==== Light Elements =====		==== Noble Elements =====			===== Lanthanides =======		
4 Be		44 Ru	<		57+La	<2e	0.004
5 B		45 Rh	<		58 Ce	<	
6 C		46 Pd	<		59 Pr	<	
7 N		47+Ag	1.49	0.06	60 Nd	<	
8 O		75+Re	0.021	0.008	62 Sm	<	
9 F	<	76 Os	<		63 Eu	<2e	0.002
		77+Ir	0.026	0.008	64 Gd	<	
		78 Pt	<		65 Tb	<2e	0.002
		79+Au	1.37	0.05	66 Dy	<	
					67 Ho	<	
					68 Er	<	
					69 Tm	<	
					70 Yb	<	
					71+Lu	<	

```
KnownConc= 0                  REST= 0              D/S= 0
Sum Conc's before normalisation to 100% :   100.1 %
```

Table 19

```
Quantum Catalytics, LLC          ANALYSIS REPORT                by Uniquant
--------------------------------------------------------------------------------
OLD.331 of  4-Jun-01                                    Today  4-Jun-01
Spectrometers configuration:  ARL 8410 Rh 60kV LiF220 LiF420 Ge111 TlAP
Sample ident   =  14-01-06 Cu/ Ag/Au RADIAL
Further info   =  14-01-06 Cu/Ag/Au RADIAL 06/04/01
Kappa list     =  15-Nov-94    Channel list =   4-Jun-01
Calculated as  :  Elements     Spectral impurity data : CAL.909 Teflon (7/94)
X-ray path     =  Vacuum       Film type    = No supporting film
Case number    =  0   Known Area, %Rest, Diluent/Sample and Mass/Area
Eff.Diam.      = 25.00 mm       Eff.Area     = 490.6 mm2
KnownConc      =  0    %
Rest           =  0    %
Dil/Sample     =  0
Viewed Mass    = 18000.00 mg
Sample Height  =  5  mm
```

```
             <     means that the concentration is <  20 ppm
             <2e   means wt% < 2 StdErr. The + in Z+El means involved in Sum=100%
   Z        wt%    StdErr    Z       wt%    StdErr    Z        wt%    StdErr
========================    ========================    ========================
SumBe..F   0      0.040    29+Cu    97.04  0.08      51 Sb      <
11+Na      0.045  0.010    30+Zn    <                52 Te      <
12 Mg      <2e    0.004    31 Ga    <2e    0.003     53 I       <
13+Al      0.047  0.004    32 Ge    <2e    0.002     55 Cs      <2e    0.003
14 Si      <2e    0.005    33 As    <                56 Ba      <2e    0.003

15+P       <               34+Se    <                SumLa..Lu  0.003  0.071
16+S       0.0066 0.0006   35+Br    <                72+Hf      <
16 So                      37+Rb    <                73+Ta      <
17+Cl      0.025  0.002    38 Sr    <                74 W       <
18+Ar      0.011  0.002    39 Y     <                75 Re      <2e    0.008

19+K       0.0021 0.0008   40+Zr    0.008  0.001     76 Os      <
20+Ca      0.0060 0.0009   41 Nb    <                77+Ir      0.033  0.008
21 Sc      <               42 Mo    <                78 Pt      <
22 Ti      <               44 Ru    <                79+Au      1.33   0.05
23 V       <               45 Rh    <                80 Hg      <2e    0.006

24+Cr      <               46 Pd    <                81 Tl      <
25 Mn      <               47+Ag    1.44   0.05      82 Pb      <
26 Fe      <               48 Cd    <                83 Bi      <
27 Co      <               49 In    <                90 Th      <
28 Ni      <               50+Sn    <                92 U       <

==== Light Elements =====  ==== Noble Elements =====  ===== Lanthanides =======
 4 Be                      44 Ru    <                57 La      <
 5 B                       45 Rh    <                58 Ce      <
 6 C                       46 Pd    <                59 Pr      <
 7 N                       47+Ag    1.44   0.05      60 Nd      <
 8 O                       75 Re    <2e    0.008     62 Sm      <
 9 F       <               76 Os    <                63 Eu      <
                           77+Ir    0.033  0.008     64 Gd      <
                           78 Pt    <                65 Tb      <2e    0.002
                           79+Au    1.33   0.05      66 Dy      <
                                                     67 Ho      <
                                                     68 Er      <
                                                     69 Tm      <
                                                     70 Yb      <
                                                     71+Lu      <
```

```
KnownConc= 0                    REST= 0                  D/S= 0
Sum Conc's before normalisation to 100% :  100.1 %
```

Table 20

```
Quantum Catalytics, LLC          ANALYSIS REPORT                by Uniquant
-----------------------------------------------------------------------------
OLD.336 of 11-Jun-01                                       Today 11-Jun-01
Spectrometers configuration: ARL 8410 Rh 60kV LiF220 LiF420 Ge111 TlAP
Sample ident   =  14-01-07     AXIAL
Further info   =  14-01-07 AXIAL LEAD TIN ZINC 6/11 CASS 8
Kappa list     =  15-Nov-94    Channel list =    4-Jun-01
Calculated as  :  Elements     Spectral impurity data : CAL.909 Teflon (7/94)
X-ray path     =  Vacuum       Film type    = No supporting film
Case number    =  0  Known Area, %Rest, Diluent/Sample and Mass/Area
Eff.Diam.      = 25.00 mm       Eff.Area      = 490.6 mm2
KnownConc      =  0    %
Rest           =  0    %
Dil/Sample     =  0
Viewed Mass    = 18000.00 mg
Sample Height  =  5   mm
              <      means that the concentration is <  20 ppm
              <2e  means wt% < 2 StdErr. The + in Z+El means involved in Sum=100%
```

Z	wt%	StdErr	Z	wt%	StdErr	Z	wt%	StdErr
SumBe..F	0	0.051	29+Cu	0.24	0.02	51+Sb	0.023	0.006
11+Na	<		30+Zn	25.4	0.2	52 Te	<	
12+Mg	0.016	0.008	31+Ga	<		53+I	<2e	0.82
13+Al	2.08	0.07	32 Ge	<		55 Cs	<	
14+Si	0.018	0.004	33 As	<		56 Ba	<	
15+P	<		34 Se	<		SumLa..Lu	0.035	0.072
16 S			35 Br	<2e	0.001	72 Hf	<	
16+So	0.038	0.003	37+Rb	<		73 Ta	<	
17+Cl	0.053	0.004	38 Sr	<		74+W	<2e	0.012
18 Ar	<		39+Y	<2e	0.002	75 Re	<	
19 K	<		40+Zr	0.024	0.005	76+Os	<	
20+Ca	<2e	0.031	41 Nb	<		77+Ir	<	
21 Sc	<		42 Mo	<		78+Pt	<	
22 Ti	<		44+Ru	<2e	0.005	79 Au	<	
23 V	<		45 Rh	<		80 Hg	<	
24 Cr	<		46 Pd	<		81+Tl	<	
25 Mn	<		47 Ag	<		82+Pb	16.9	0.2
26 Fe	<		48+Cd	0.014	0.005	83+Bi	<	
27 Co	<2e	0.001	49+In	<2e	0.011	90+Th	<	
28+Ni	0.007	0.003	50+Sn	54.7	0.2	92 U	<	

==== Light Elements =====		==== Noble Elements ====			===== Lanthanides =======		
4 Be		44+Ru	<2e	0.005	57 La	<	
5 B		45 Rh	<		58+Ce	0.030	0.010
6 C		46 Pd	<		59 Pr	<2e	0.006
7 N		47 Ag	<		60+Nd	<2e	0.004
8 O		75 Re	<		62 Sm	<	
9 F	<	76+Os	<		63 Eu	<	
		77+Ir	<		64 Gd	<	
		78+Pt	<		65 Tb	<	
		79 Au	<		66 Dy	<	
					67 Ho	<	
					68 Er	<	
					69 Tm	<	
					70 Yb	<	
					71 Lu	<	

```
KnownConc= 0                    REST= 0              D/S= 0
Sum Conc's before normalisation to 100% :   111.4 %
```

Table 21

```
Quantum Catalytics, LLC          ANALYSIS REPORT              by Uniquant
-------------------------------------------------------------------------
OLD.337 of  4-Jun-01                                      Today 11-Jun-01
Spectrometers configuration:  ARL 8410 Rh 60kV LiF220 LiF420 Ge111 TlAP
Sample ident  =  14-01-07     RADIAL
Further info  =  14-01-07 RADIAL LEAD/TIN/ZINC 6/11  CS 8
Kappa list    =  15-Nov-94    Channel list  =   4-Jun-01
Calculated as :  Elements     Spectral impurity data : CAL.909 Teflon (7/94)
X-ray path    =  Vacuum       Film type     = No supporting film
Case number   =  0    Known Area, %Rest, Diluent/Sample and Mass/Area
Eff.Diam.     =  25.00 mm      Eff.Area      = 490.6 mm2
KnownConc     =  0     %
Rest          =  0     %
Dil/Sample    =  0
Viewed Mass   =  18000.00 mg
Sample Height =  5   mm
              <      means that the concentration is <  20 ppm
              <2e  means wt% < 2 StdErr. The + in Z+El means involved in Sum=100%
```

Z	wt%	StdErr	Z	wt%	StdErr	Z	wt%	StdErr
SumBe..F	0	0.046	29+Cu	0.014	0.002	51+Sb	0.015	0.005
11+Na	<		30+Zn	33.2	0.2	52 Te	<	
12+Mg	<		31+Ga	<		53+I	<2e	0.72
13+Al	0.17	0.01	32 Ge	<		55 Cs	<	
14+Si	0.026	0.004	33 As	<		56 Ba	<	
15 P	<		34 Se	<		SumLa..Lu	0.031	0.069
16 S			35 Br	<		72 Hf	<	
16+So	<		37+Rb	<		73 Ta	<	
17+Cl	0.031	0.003	38 Sr	<		74+W	0.033	0.014
18 Ar	<		39+Y	<		75 Re	<	
19 K	<		40+Zr	0.016	0.005	76+Os	<	
20+Ca	<2e	0.027	41 Nb	<		77+Ir	<	
21 Sc	<		42 Mo	<		78+Pt	<	
22 Ti	<		44+Ru	0.011	0.005	79 Au	<	
23 V	<		45 Rh	<		80 Hg	<	
24 Cr	<		46 Pd	<		81+Tl	<2e	0.014
25 Mn	<		47 Ag	<		82+Pb	18.3	0.2
26 Fe	<		48+Cd	0.025	0.004	83 Bi	<	
27 Co	<		49+In	<2e	0.010	90 Th	<	
28 Ni	<		50+Sn	47.9	0.2	92 U	<	

==== Light Elements =====			==== Noble Elements =====			===== Lanthanides ======		
4 Be			44+Ru	0.011	0.005	57 La	<2e	0.008
5 B			45 Rh	<		58+Ce	<2e	0.010
6 C			46 Pd	<		59 Pr	<	
7 N			47 Ag	<		60 Nd	<	
8 O			75 Re	<		62 Sm	<	
9 F	<		76+Os	<		63 Eu	<	
			77+Ir	<		64 Gd	<2e	0.004
			78+Pt	<		65 Tb	<	
			79 Au	<		66 Dy	<	
						67+Ho	0.012	0.006
						68 Er	<2e	0.004
						69 Tm	<2e	0.004
						70 Yb	<2e	0.004
						71 Lu	<	

```
KnownConc= 0                    REST= 0                    D/S= 0
Sum Conc's before normalisation to 100% :  115.8 %
```

Table 22

```
Quantum Catalytics, LLC          ANALYSIS REPORT              by Uniquant
------------------------------------------------------------------------
OLD.342 of 14-Jun-01                               Today 14-Jun-01
Spectrometers configuration:  ARL 8410 Rh 60kV LiF220 LiF420 Ge111 TlAP
Sample ident   =  14-01-08     AXIAL
Further info   = .14-01-08 AXIAL Sn,K,Na,Mg 6/14
Kappa list     =  15-Nov-94    Channel list =   4-Jun-01
Calculated as  :  Elements     Spectral impurity data : CAL.909 Teflon (7/94)
X-ray path     =  Vacuum       Film type    = No supporting film
Case number    =  0  Known Area, %Rest, Diluent/Sample and Mass/Area
Eff.Diam.      = 25.00 mm       Eff.Area     = 490.6 mm2
KnownConc      =  0     %
Rest           =  0     %
Dil/Sample     =  0
Viewed Mass    = 18000.00 mg
Sample Height  =  5   mm
```

```
          <     means that the concentration is <  20 ppm
          <2e   means wt% < 2 StdErr. The + in Z+El means involved in Sum=100%
   Z      wt%    StdErr    Z        wt%    StdErr     Z       wt%    StdErr
========================  ========================  =========================
SumBe..F  0      0.060     29+Cu   0.079  0.006      51 Sb    <
11+Na     1.07   0.05      30 Zn   <                 52 Te    <
12+Mg     0.85   0.04      31 Ga   <                 53+I     <2e    1.43
13+Al     1.53   0.06      32 Ge   <                 55 Cs    <
14+Si     0.16   0.01      33 As   <                 56 Ba    <

15+P      <                34 Se   <                 SumLa..Lu 0.021 0.072
16 S                       35 Br   <                 72 Hf    <2e    0.003
16+So     0.044  0.004     37 Rb   <                 73 Ta    <
17+Cl     0.015  0.001     38+Sr   <                 74+W     0.031  0.004
18 Ar     <                39 Y    <                 75 Re    <

19 K      <                40+Zr   0.047  0.004      76 Os    <
20+Ca     <                41+Nb   <                 77+Ir    <2e    0.003
21+Sc     0.007  0.002     42 Mo   <                 78 Pt    <
22 Ti     <                44+Ru   <                 79 Au    <
23 V      <2e    0.002     45 Rh   <                 80 Hg    <

24 Cr     <                46 Pd   <                 81 Tl    <
25 Mn     <                47 Ag   <                 82+Pb    0.067  0.006
26+Fe     0.017  0.002     48+Cd   0.041  0.005      83 Bi    <2e    0.002
27+Co     0.003  0.001     49+In   <2e    0.019      90+Th    <
28+Ni     0.021  0.002     50+Sn   95.6   0.1        92 U     <

==== Light Elements =====  ==== Noble Elements =====  ===== Lanthanides =======
   4 Be                    44+Ru   <           -      57 La    <
   5 B                     45 Rh   <                  58 Ce    <2e    0.007
   6 C                     46 Pd   <                  59 Pr    <
   7 N                     47 Ag   <                  60 Nd    <2e    0.005
   8 O                     75 Re   <                  62 Sm    <
   9 F      <              76 Os   <                  63 Eu    <
                           77+Ir   <2e    0.003       64 Gd    <
                           78 Pt   <                  65 Tb    <
                           79 Au   <                  66 Dy    <
                                                      67 Ho    <2e    0.005
                                                      68 Er    <
                                                      69 Tm    <2e    0.004
                                                      70 Yb    <
                                                      71 Lu    <
```

```
KnownConc= 0                    REST= 0                    D/S= 0
Sum Conc's before normalisation to 100% :    94.8 %
```

Table 23

```
Quantum Catalytics, LLC          ANALYSIS REPORT              by Uniquant
-------------------------------------------------------------------------
OLD.343 of  4-Jun-01                                  Today 14-Jun-01
Spectrometers configuration:  ARL 8410 Rh 60kV LiF220 LiF420 Ge111 TlAP
Sample ident  =  14-01-08     RADIAL
Further info  =  14-01-08 RADIAL Sn,K,Na,Mg 6/14
Kappa list    =  15-Nov-94    Channel list =   4-Jun-01
Calculated as :  Elements     Spectral impurity data : CAL.909 Teflon (7/94)
X-ray path    =  Vacuum       Film type    = No supporting film
Case number   =  0   Known Area, %Rest, Diluent/Sample and Mass/Area
Eff.Diam.     =  25.00 mm      Eff.Area     = 490.6 mm2
KnownConc     =  0    %
Rest          =  0    %
Dil/Sample    =  0
Viewed Mass   =  18000.00 mg
Sample Height =  5   mm
             <    means that the concentration is <  20 ppm
            <2e   means wt% < 2 StdErr. The + in Z+El means involved in Sum=100%
```

Z	wt%	StdErr	Z	wt%	StdErr	Z	wt%	StdErr
SumBe..F	0	0.056	29+Cu	0.14	0.01	51 Sb	<	
11+Na	2.82	0.08	30+Zn	0.003	0.002	52 Te	<	
12+Mg	1.04	0.04	31 Ga	<		53+I	<2e	1.40
13+Al	1.69	0.06	32 Ge	<		55 Cs	<	
14+Si	0.29	0.02	33 As	<		56 Ba	<	
15+P	0.0022	0.0004	34 Se	<		SumLa..Lu	0.025	0.065
16 S			35 Br	<		72 Hf	<2e	0.003
16+So	0.032	0.003	37 Rb	<		73 Ta	<	
17+Cl	0.013	0.001	38 Sr	<		74+W	0.048	0.004
18 Ar	<		39 Y	<		75 Re	<	
19 K	<		40+Zr	0.043	0.004	76 Os	<2e	0.003
20+Ca	<		41+Nb	<		77+Ir	<2e	0.003
21 Sc	<2e	0.002	42 Mo	<		78 Pt	<	
22 Ti	<		44+Ru	<		79 Au	<	
23 V	<2e	0.001	45 Rh	<		80 Hg	<	
24 Cr	<2e	0.002	46 Pd	<		81+Tl	0.006	0.003
25 Mn	<2e	0.001	47 Ag	<		82+Pb	0.069	0.006
26+Fe	0.023	0.002	48+Cd	0.029	0.006	83 Bi	<2e	0.002
27 Co	<2e	0.001	49+In	<2e	0.019	90+Th	<.	
28+Ni	0.045	0.004	50+Sn	93.6	0.1	92 U	<	

==== Light Elements =====			==== Noble Elements =====			===== Lanthanides =======		
4 Be			44+Ru	<		57 La	<	
5 B			45 Rh	<		58 Ce	<2e	0.006
6 C			46 Pd	<		59 Pr	<	
7 N			47 Ag	<		60 Nd	<2e	0.004
8 O			75 Re	<		62 Sm	<2e	0.005
9 F	<		76 Os	<2e	0.003	63 Eu	<2e	0.004
			77+Ir	<2e	0.003	64 Gd	<	
			78 Pt	<		65 Tb	<	
			79 Au	<		66 Dy	<	
						67 Ho	<	
						68 Er	<	
						69 Tm	<	
						70 Yb	<	
						71 Lu	<	

```
KnownConc= 0                        REST= 0                  D/S= 0
Sum Conc's before normalisation to 100% :   98.1 %
```

Table 24

```
Quantum Catalytics, LLC          ANALYSIS REPORT                by Uniquant
------------------------------------------------------------------------------
OLD.314 of 24-May-01                                   Today 24-May-01
Spectrometers configuration:  ARL 8410 Rh 60kV LiF220 LiF420 Ge111 TlAP
Sample ident  =  15-01-01 Si  AXIAL BLK
Further info  =  15-01-01 Si  AXIAL BLK 5/24
Kappa list    =  15-Nov-94    Channel list =  24-May-01
Calculated as :  Elements    Spectral impurity data : CAL.909 Teflon (7/94)
X-ray path    =  Vacuum       Film type    = No supporting film
Case number   =  0   Known Area, %Rest, Diluent/Sample and Mass/Area
Eff.Diam.     = 25.00 mm       Eff.Area     = 490.6 mm2
KnownConc     =  0     %
Rest          =  0     %
Dil/Sample    =  0
Viewed Mass   = 18000.00 mg
Sample Height =  5   mm
              <   means that the concentration is <  20 ppm
              <2e means wt% < 2 StdErr. The + in Z+El means involved in Sum=100%
   Z     wt%     StdErr    Z           wt%   StdErr    Z            wt%    StdErr
 ========================  ========================  ==========================
 SumBe..F  0.059   0.021   29+Cu     0.012  0.001   51 Sb        <
 11+Na     0.004   0.002   30+Zn     0.0021 0.0005  52 Te        <
 12 Mg     <               31+Ga     0.0038 0.0005  53 I         <
 13+Al     1.18    0.05    32 Ge     <               55+Cs       <2e    0.003
 14+Si    98.58    0.06    33 As     <               56+Ba       0.008  0.003

 15+P      0.013   0.001   34 Se     <               SumLa..Lu   0.004  0.018
 16+S      0.013   0.001   35 Br     <               72 Hf       <
 16 So                     37 Rb     <               73 Ta       <
 17+Cl     0.004   0.002   38 Sr     <               74+W        0.007  0.001
 18+Ar     0.013   0.003   39 Y      <               75 Re       <

 19+K      <               40+Zr     <               76 Os       <
 20+Ca     0.0092  0.0009  41 Nb     <               77 Ir       <
 21 Sc     <               42 Mo     <               78 Pt       <
 22+Ti     0.019   0.002   44 Ru     <               79 Au       <
 23 V      <               45 Rh     <               80 Hg       <

 24+Cr     0.0031  0.0005  46 Pd     <               81 Tl       <
 25+Mn     <               47 Ag     <               82 Pb       <
 26+Fe     0.055   0.005   48 Cd     <               83 Bi       <
 27 Co     <               49 In     <               90 Th       <
 28+Ni     0.0051  0.0006  50 Sn     <               92 U        <

 ==== Light Elements =====  ==== Noble Elements =====  ===== Lanthanides =======
    4 Be                      44 Ru     <              57 La       <
    5 B                       45 Rh     <              58 Ce       <
    6 C                       46 Pd     <              59 Pr       <
    7 N                       47 Ag     <              60 Nd       <
    8 O                       75 Re     <              62 Sm       <
    9+F     0.059   0.021     76 Os     <              63 Eu       <
                             77 Ir     <              64 Gd       <
                             78 Pt     <              65 Tb       <
                             79 Au     <              66 Dy       <
                                                      67 Ho       <
                                                      68 Er       <
                                                      69 Tm       <
                                                      70 Yb       <
                                                      71 Lu       <

 KnownConc= 0                    REST= 0                  D/S= 0
 Sum Conc's before normalisation to 100% :    83.4 %
```

Table 25

```
Quantum Catalytics, LLC              ANALYSIS REPORT
-------------------------------------------------------------------------------
OLD.317 of 24-May-01                                      Today 24-May·
Spectrometers configuration:  ARL 8410 Rh 60kV LiF220 LiF420 Ge111 T1AP
Sample ident  =  15-01-01 Si  RADIAL BLK
Further info  =  15-01-01 Si RADIAL BLK 5/24
Kappa list    =  15-Nov-94    Channel list =  24-May-01
Calculated as :  Elements     Spectral impurity data : CAL.909 Teflon (7/94)
X-ray path    =  Vacuum       Film type    = No supporting film
Case number   =  0   Known Area, %Rest, Diluent/Sample and Mass/Area
Eff.Diam.     =  25.00 mm      Eff.Area     =  490.6 mm2
KnownConc     =  0    %
Rest          =  0    %
Dil/Sample    =  0
Viewed Mass   =  18000.00 mg
Sample Height =  5   mm
           <     means that the concentration is <  20 ppm
           <2e  means wt% < 2 StdErr. The + in Z+El means involved in Sum=100%
```

Z	wt%	StdErr	Z	wt%	StdErr	Z	wt%	StdErr
SumBe..F	0	0.021	29+Cu	0.0101	0.0009	51 Sb	<	
11+Na	0.016	0.002	30+Zn	0.0020	0.0005	52 Te	<	
12 Mg	<		31+Ga	0.0043	0.0005	53 I	<	
13+Al	1.46	0.05	32 Ge	<		55+Cs	<2e	0.003
14+Si	98.12	0.07	33 As	<		56+Ba	<2e	0.003
15+P	0.018	0.002	34 Se	<		SumLa..Lu	0.005	0.020
16 S			35 Br	<		72+Hf	0.003	0.001
16+So	0.14	0.01	37 Rb	<		73 Ta	<	
17+Cl	0.019	0.002	38 Sr	<		74+W	0.011	0.001
18+Ar	0.012	0.003	39 Y	<		75 Re	<	
19+K	0.014	0.001	40+Zr	<		76 Os	<	
20+Ca	0.077	0.006	41 Nb	<		77+Ir	<	
21 Sc	<		42 Mo	<		78 Pt	<	
22+Ti	0.029	0.003	44 Ru	<		79 Au	<	
23+V	<		45 Rh	<		80 Hg	<	
24+Cr	0.0026	0.0005	46 Pd	<		81 Tl	<	
25 Mn	<		47 Ag	<		82 Pb	<	
26+Fe	0.042	0.004	48 Cd	<		83 Bi	<	
27 Co	<		49 In	<		90 Th	<	
28+Ni	0.0053	0.0006	50 Sn	<		92 U	<	

```
==== Light Elements =====    ==== Noble Elements =====   ===== Lanthanides =======
  4 Be                        44 Ru   <                  57 La   <
  5 B                         45 Rh   <                  58 Ce   <
  6 C                         46 Pd   <                  59 Pr   <
  7 N                         47 Ag   <                  60 Nd   <
  8 O                         75 Re   <                  62 Sm   <
  9 F      <                  76 Os   <                  63 Eu   <
                              77+Ir   <                  64 Gd   <2e     0.001
                              78 Pt   <                  65 Tb   <
                              79 Au   <                  66 Dy   <
                                                         67 Ho   <
                                                         68 Er   <
                                                         69 Tm   <
                                                         70 Yb   <
                                                         71 Lu   <

KnownConc= 0                     REST= 0                    D/S= 0
Sum Conc's before normalisation to 100% :   79.7 %
```

Table 26

```
Quantum Catalytics, LLC          ANALYSIS REPORT                 by Uniquant
----------------------------------------------------------------------------
OLD.237 of 23-Apr-01                                    Today 23-Apr-01
Spectrometers configuration:  ARL 8410 Rh 60kV LiF220 LiF420 Ge111 TlAP
Sample ident  =  15-01-01 Si  SECTION TOP
Further info  =  15-01-01 Si MID SECTION TOP FACE AXIAL
Kappa list    =  15-Nov-94    Channel list = 19-Apr-01
Calculated as :  Elements     Spectral impurity data : CAL.909 Teflon (7/94)
X-ray path    =  Vacuum       Film type     = No supporting film
Case number   =  0   Known Area, %Rest, Diluent/Sample and Mass/Area
Eff.Diam.     =  25.00 mm     Eff.Area      = 490.6 mm2
KnownConc     =  0    %
Rest          =  0    %
Dil/Sample    =  0
Viewed Mass   =  18000.00 mg
Sample Height =  5   mm
              <    means that the concentration is < 20 ppm
              <2e  means wt% < 2 StdErr. The + in Z+El means involved in Sum=100%
```

Z	wt%	StdErr	Z	wt%	StdErr	Z	wt%	StdErr
SumBe..F	0	0.018	29+Cu	0.0070	0.0006	51 Sb	<	
11+Na	0.050	0.004	30+Zn	0.0033	0.0005	52 Te	<	
12 Mg	<		31+Ga	0.0020	0.0004	53 I	<	
13+Al	1.02	0.04	32 Ge	<		55 Cs	<2e	0.003
14+Si	98.48	0.06	33 As	<		56+Ba	<2e	0.003
15+P	0.019	0.002	34 Se	<		SumLa..Lu	0.002	0.018
16+S	0.14	0.01	35 Br	<		72 Hf	<	
16 So			37 Rb	<		73 Ta	<	
17+Cl	0.075	0.006	38 Sr	<		74+W	0.009	0.001
18+Ar	0.012	0.003	39 Y	<		75 Re	<	
19+K	0.028	0.002	40+Zr	<		76 Os	<	
20+Ca	0.025	0.002	41 Nb	<		77 Ir	<	
21 Sc	<		42 Mo	<		78 Pt	<	
22+Ti	0.064	0.005	44 Ru	<		79 Au	<	
23+V	<		45 Rh	<		80 Hg	<	
24+Cr	<		46 Pd	<		81 Tl	<	
25 Mn	<		47 Ag	<		82 Pb	<	
26+Fe	0.045	0.004	48+Cd	<		83 Bi	<	
27+Co	<		49 In	<		90+Th	<2e	0.001
28+Ni	0.0051	0.0006	50 Sn	<		92 U	<	

```
==== Light Elements =====    ==== Noble Elements =====   ===== Lanthanides =======
 4 Be                        44 Ru       <               57 La       <
 5 B                         45 Rh       <               58 Ce       <
 6 C                         46 Pd       <               59 Pr       <
 7 N                         47 Ag       <               60 Nd       <
 8 O                         75 Re       <               62 Sm       <
 9 F        <                76 Os       <               63 Eu       <
                             77 Ir       <               64 Gd       <
                             78 Pt       <               65 Tb       <
                             79 Au       <               66 Dy       <
                                                         67 Ho       <
                                                         68 Er       <
                                                         69 Tm       <
                                                         70 Yb       <
                                                         71 Lu       <

KnownConc= 0                      REST= 0                    D/S= 0
Sum Conc's before normalisation to 100% :    90.0 %
```

Table 27

```
Quantum Catalytics, LLC          ANALYSIS REPORT              by Uniquant
------------------------------------------------------------------------
OLD.245 of 19-Apr-01                                    Today 24-Apr-01
Spectrometers configuration: ARL 8410 Rh 60kV LiF220 LiF420 Ge111 TlAP
Sample ident    =  15-01-01 MID BOTTOM FACE
Further info    =  15-01-01 Si MID SECTION BOT FACE AXIAL
Kappa list      =  15-Nov-94    Channel list = 19-Apr-01
Calculated as  :  Elements     Spectral impurity data : CAL.909 Teflon (7/94)
X-ray path      =  Vacuum       Film type    = No supporting film
Case number     =  0  Known Area, %Rest, Diluent/Sample and Mass/Area
Eff.Diam.       =  25.00 mm     Eff.Area     = 490.6 mm2
KnownConc       =  0   %
Rest            =  0   %
Dil/Sample      =  0
Viewed Mass     =  18000.00 mg
Sample Height  =  5  mm
              <     means that the concentration is < 20 ppm
              <2e   means wt% < 2 StdErr. The + in Z+El means involved in Sum=100%
   Z        wt%     StdErr     Z        wt%    StdErr      Z         wt%    StdErr
========================== ========================== ==========================
SumBe..F    0       0.017    29+Cu    0.0075  0.0007   51 Sb       <
11+Na       0.033   0.003    30+Zn    <                52 Te       <
12 Mg       <                31+Ga    0.0021  0.0003   53 I        <
13+Al       0.71    0.04     32 Ge    <                55+Cs       <2e     0.002
14+Si       98.81   0.05     33 As    <                56+Ba       <2e     0.003

15+P        0.016   0.001    34 Se    <                SumLa..Lu   0.006   0.018
16+S        0.23    0.02     35 Br    <                72 Hf       <
16 So                        37 Rb    <                73 Ta       <
17+Cl       0.057   0.005    38 Sr    <                74+W        0.007   0.001
18+Ar       0.007   0.002    39 Y     <                75 Re       <

19+K        0.012   0.001    40 Zr    <                76 Os       <
20+Ca       0.041   0.003    41 Nb    <                77 Ir       <
21 Sc       <                42 Mo    <                78 Pt       <
22+Ti       0.020   0.002    44 Ru    <                79 Au       <
23+V        <                45 Rh    <                80 Hg       <

24+Cr       <                46 Pd    <                81 Tl       <
25 Mn       <                47 Ag    <                82 Pb       <
26+Fe       0.026   0.002    48 Cd    <                83 Bi       <
27+Co       <                49 In    <                90 Th       <
28+Ni       0.0034  0.0006   50 Sn    <                92 U        <

==== Light Elements =====  ==== Noble Elements =====  ==== Lanthanides ======
   4 Be                        44 Ru    <                57 La       <
   5 B                         45 Rh    <                58 Ce       <
   6 C                         46 Pd    <                59 Pr       <
   7 N                         47 Ag    <                60 Nd       <
   8 O                         75 Re    <                62 Sm       <
   9 F        <                76 Os    <                63 Eu       <
                              77 Ir    <                64 Gd       <
                              78 Pt    <                65 Tb       <
                              79 Au    <                66 Dy       <
                                                        67 Ho       <2e     0.002
                                                        68+Er       <2e     0.001
                                                        69 Tm       <
                                                        70 Yb       <
                                                        71 Lu       <

KnownConc= 0                    REST= 0                   D/S= 0
Sum Conc's before normalisation to 100% :    91.9 %
```

Table 28

```
Quantum Catalytics, LLC          ANALYSIS REPORT               by Uniquant
-----------------------------------------------------------------------------
OLD.326 of  1-Jun-01                                    Today  4-Jun-01
Spectrometers configuration:  ARL 8410 Rh 60kV LiF220 LiF420 Ge111 T1AP
Sample ident  =  15-01-02      AXIAL TOP
Further info  =  15-01-02 AXIAL TOP
Kappa list    =  15-Nov-94      Channel list = 24-May-01
Calculated as :  Elements       Spectral impurity data : CAL.909 Teflon (7/94)
X-ray path    =  Vacuum         Film type    = No supporting film
Case number   =  0   Known Area, %Rest, Diluent/Sample and Mass/Area
Eff.Diam.     = 25.00 mm        Eff.Area     = 490.6 mm2
KnownConc     =  0    %
Rest          =  0    %
Dil/Sample    =  0
Viewed Mass   = 18000.00 mg
Sample Height =  5  mm
              <      means that the concentration is <  20 ppm
              <2e  means wt% < 2 StdErr. The + in Z+El means involved in Sum=100%
```

Z	wt%	StdErr	Z	wt%	StdErr	Z	wt%	StdErr
SumBe..F	0	0.026	29+Cu	0.012	0.002	51 Sb	<	
11 Na	<2e	0.009	30 Zn	<		52 Te	<	
12 Mg	<		31 Ga	<		53 I	<	
13+Al	1.89	0.06	32 Ge	<		55 Cs	<	
14+Si	0.032	0.005	33 As	<		56 Ba	<2e	0.003
15+P	0.0033	0.0003	34 Se	<		SumLa..Lu	0	0.20
16 S			35 Br	<		72 Hf	<2e	0.005
16+So	0.0077	0.0007	37 Rb	<		73 Ta	<	
17+Cl	0.0040	0.0006	38 Sr	<		74+W	0.009	0.004
18+Ar	0.012	0.001	39 Y	<		75 Re	<2e	0.004
19 K	<		40+Zr	0.0099	0.0009	76 Os	<	
20+Ca	0.0053	0.0006	41 Nb	<		77 Ir	<	
21+Sc	0.0025	0.0005	42 Mo	<		78 Pt	<	
22 Ti	<		44 Ru	<		79 Au	<	
23 V	<		45 Rh	<		80 Hg	<	
24 Cr	<		46 Pd	<		81 Tl	<	
25 Mn	<		47 Ag	<2e	0.002	82 Pb	<	
26+Fe	98.01	0.07	48 Cd	<2e	0.001	83 Bi	<	
27 Co	<		49 In	<		90+Th	0.006	0.002
28 Ni	<		50 Sn	<		92 U	<	

==== Light Elements =====		==== Noble Elements =====			===== Lanthanides ======		
4 Be		44 Ru	<		57 La	<	
5 B		45 Rh	<		58 Ce	<	
6 C		46 Pd	<		59+Pr	<2e	0.006
7 N		47 Ag	<2e	0.002	60 Nd	<	
8 O		75 Re	<2e	0.004	62+Sm	<	
9 F	<	76 Os	<		63 Eu	<	
		77 Ir	<		64 Gd	<	
		78 Pt	<		65+Tb	<	
		79 Au	<		66 Dy	<	
					67 Ho	<	
					68+Er	<	
					69+Tm	<	
					70 Yb	<	
					71 Lu	<	

```
KnownConc= 0                    REST= 0              D/S= 0
Sum Conc's before normalisation to 100% :   96.5 %
```

Table 29

```
Quantum Catalytics, LLC          ANALYSIS REPORT               by Uniquant
------------------------------------------------------------------------------
OLD.329 of  1-Jun-01                               Today  4-Jun-01
Spectrometers configuration:  ARL 8410 Rh 60kV LiF220 LiF420 Ge111 TlAP
Sample ident  =  15-01-02 Fe  RADIAL
Further info  =  15-01-02 Fe  RADIAL
Kappa list    =  15-Nov-94    Channel list = 24-May-01
Calculated as :  Elements     Spectral impurity data : CAL.909 Teflon (7/94)
X-ray path    =  Vacuum       Film type    = No supporting film
Case number   =  0   Known Area, %Rest, Diluent/Sample and Mass/Area
Eff.Diam.     =  25.00 mm     Eff.Area     = 490.6 mm2
KnownConc     =  0     %
Rest          =  0     %
Dil/Sample    =  0
Viewed Mass   =  18000.00 mg
Sample Height =  5   mm
```

	< means that the concentration is < 20 ppm
	<2e means wt% < 2 StdErr. The + in Z+El means involved in Sum=100%

Z	wt%	StdErr	Z	wt%	StdErr	Z	wt%	StdErr
SumBe..F	0	0.024	29+Cu	0.014	0.002	51 Sb	<	
11+Na	0.040	0.009	30 Zn	<		52 Te	<	
12 Mg	<		31 Ga	<		53 I	<	
13+Al	2.96	0.08	32 Ge	<		55 Cs	<	
14+Si	0.14	0.01	33 As	<		56 Ba	<	
15+P	0.0036	0.0003	34 Se	<		SumLa..Lu	0	0.20
16 S			35 Br	<		72 Hf	<	
16+So	0.012	0.001	37 Rb	<		73 Ta	<	
17+Cl	0.0044	0.0006	38 Sr	<		74 W	<	
18+Ar	0.013	0.001	39 Y	<		75 Re	<2e	0.004
19 K	<		40+Zr	0.012	0.001	76 Os	<	
20+Ca	0.0073	0.0007	41 Nb	<		77 Ir	<	
21+Sc	0.0026	0.0005	42 Mo	<		78 Pt	<	
22 Ti	<		44 Ru	<		79 Au	<	
23 V	<		45 Rh	'		80 Hg	<2e	0.003
24 Cr	<		46 Pd	<		81 Tl	<	
25 Mn	<		47 Ag	<		82 Pb	<	
26+Fe	96.78	0.09	48 Cd	<		83 Bi	<	
27 Co	<		49 In	<		90 Th	<2e	0.002
28 Ni	<		50 Sn	<		92 U	<	

==== Light Elements =====	==== Noble Elements =====	===== Lanthanides =======
4 Be	44 Ru	<	57 La	<
5 B	45 Rh	<	58 Ce	<2e	0.003
6 C	46 Pd	<	59 Pr	<
7 N	47 Ag	<	60 Nd	<
8 O	75 Re	<2e	0.004	62+Sm	<
9 F	<	76 Os	<	63 Eu	<2e	0.003
	77 Ir	<	64 Gd	<
	78 Pt	<	65+Tb	<
	79 Au	<	66 Dy	<
		67 Ho	<
		68+Er	<
		69+Tm	<
		70 Yb	<
		71 Lu	<

```
KnownConc= 0                   REST= 0.              D/S= 0
Sum Conc's before normalisation to 100% :    97.8 %
```

Table 30

```
Quantum Catalytics, LLC            ANALYSIS REPORT ·                    by Uniquant
------------------------------------------------------------------------------------
OLD.338 of 11-Jun-01                                            Today 11-Jun-01
Spectrometers configuration:  ARL 8410 Rh 60kV LiF220 LiF420 Ge111 TlAP
Sample ident    =  15-01-04       AXIAL
Further info    =  15-01-04 Ni/W/Ta/Hf AXIAL 6/11
Kappa list      =  15-Nov-94      Channel list =   4-Jun-01
Calculated as :   Elements        Spectral impurity data : CAL.909 Teflon (7/94)
X-ray path      =  Vacuum         Film type    = No supporting film
Case number     =  0   Known Area, %Rest, Diluent/Sample and Mass/Area
Eff.Diam.       = 25.00 mm        Eff.Area     = 490.6 mm2
KnownConc       =  0    %
Rest            =  0    %
Dil/Sample      =  0
Viewed Mass     = 18000.00 mg
Sample Height =  5   mm
            <     means that the concentration is <  20 ppm
            <2e  means wt% < 2 StdErr. The + in Z+El means involved in Sum=100%
```

Z	wt%	StdErr	Z	wt%	StdErr	Z	wt%	StdErr
SumBe..F	0	0.036	29 Cu	<		51 Sb	<	
11+Na	0.066	0.009	30 Zn	<		52 Te	<	
12 Mg	<		31 Ga	<		53 I	<	
13+Al	3.57	0.09	32 Ge	<		55 Cs	<	
14+Si	0.23	0.02	33 As	<		56 Ba	<	
15+P	0.0025	0.0004	34+Se	<		SumLa..Lu	0.58	1.17
16+S	0.0069	0.0006	35 Br	<		72+Hf	1.78	0.06
16 So			37 Rb	<		73+Ta	1.76	0.06
17+Cl	0.0044	0.0008	38 Sr	<		74+W	1.49	0.06
18+Ar	0.018	0.002	39 Y	<		75 Re	<	
19 K	<		40+Zr	<		76 Os	<	
20+Ca	0.0025	0.0008	41 Nb	<		77+Ir	<	
21+Sc	0.0036	0.0009	42 Mo	<		78 Pt	<	
22 Ti	<		44 Ru	<		79 Au	<	
23 V	<		45 Rh	<		80+Hg	<	
24 Cr	<		46 Pd	<		81 Tl	<	
25 Mn	<		47 Ag	<		82 Pb	<	
26 Fe	<		48 Cd	<		83+Bi	<	
27 Co	<		49 In	<		90 Th	<2e	0.003
28+Ni	90.5	0.1	50 Sn	<		92 U	<	

```
==== Light Elements =====    ==== Noble Elements =====    ===== Lanthanides =======
 4  Be                        44 Ru    <                   57 La    <
 5  B                         45 Rh    <                   58 Ce    <
 6  C                         46 Pd    <                   59 Pr    <
 7  N                         47 Ag    <                   60 Nd    <
 8  O                         75 Re    <                   62 Sm    <
 9  F       <                 76 Os    <                   63 Eu    <2e    0.002
                              77+Ir    <                   64 Gd    <
                              78 Pt    <                   65 Tb    <
                              79 Au    <                   66 Dy    <
                                                           67+Ho    <
                                                           68+Er    <
                                                           69+Tm    <
                                                           70+Yb    0.58   0.26
                                                           71 Lu    <


KnownConc= 0                      REST= 0                      D/S= 0
Sum Conc's before normalisation to 100% :  101.0 %
```

Table 31

```
Quantum Catalytics, LLC          ANALYSIS REPORT              by Uniquant
------------------------------------------------------------------------
OLD.339 of  4-Jun-01                                 Today 11-Jun-01
Spectrometers configuration: ARL 8410 Rh 60kV LiF220 LiF420 Ge111 TlAP
Sample ident   = 15-01-04       RADIAL
Further info   = 15-01-04 Ni/W/Ta/Hf RADIAL 6/11
Kappa list     = 15-Nov-94    Channel list =   4-Jun-01
Calculated as :  Elements      Spectral impurity data : CAL.909 Teflon (7/94)
X-ray path     = Vacuum        Film type    = No supporting film
Case number  . = 0   Known Area, %Rest, Diluent/Sample and Mass/Area
Eff.Diam.      = 25.00 mm      Eff.Area     = 490.6 mm2
KnownConc      = 0     %
Rest           = 0     %
Dil/Sample     = 0
Viewed Mass    = 18000.00 mg
Sample Height  = 5   mm
           <    means that the concentration is <  20 ppm
           <2e  means wt% < 2 StdErr. The + in Z+El means involved in Sum=100%
```

Z	wt%	StdErr	Z	wt%	StdErr	Z	wt%	StdErr
SumBe..F	0	0.036	29+Cu	<		51 Sb	<	
11+Na	0.043	0.009	30 Zn	<		52 Te	<	
12 Mg	<		31+Ga	<2e	0.002	53 I	<	
13+Al	3.38	0.09	32+Ge	<		55 Cs	<2e	0.003
14+Si	0.74	0.04	33 As	<		56 Ba	<	
15+P	<		34+Se	<		SumLa..Lu	0.57	1.16
16+S	0.0053	0.0005	35 Br	<		72+Hf	2.00	0.07
16 So			37 Rb	<		73+Ta	1.78	0.06
17+Cl	0.0059	0.0008	38 Sr	<		74+W	1.50	0.06
18+Ar	0.019	0.002	39 Y	<		75+Re	<	
19 K	<		40+Zr	<		76 Os	<	
20 Ca	<		41 Nb	<		77+Ir	<	
21+Sc	0.0020	0.0009	42 Mo	<		78 Pt	<	
22 Ti	<		44 Ru	<		79+Au	<	
23 V	<		45 Rh	<		80+Hg	<	
24 Cr	<		46 Pd	<		81 Tl	<	
25 Mn	<		47 Ag	<		82 Pb	<	
26 Fe	<		48 Cd	<2e	0.002	83+Bi	<	
27 Co	<		49 In	<		90 Th	<2e	0.003
28+Ni	90.0	0.2	50 Sn	<		92 U	<	

==== Light Elements =====		==== Noble Elements ====		==== Lanthanides =======	
4 Be		44 Ru	<	57 La	<
5 B		45 Rh	<	58 Ce	<
6 C		46 Pd	<	59 Pr	<
7 N		47 Ag	<	60 Nd	<
8 O		75+Re	<	62 Sm	<
9 F	<	76 Os	<	63 Eu	<
		77+Ir	<	64 Gd	<2e 0.002
		78 Pt	<	65 Tb	<
		79+Au	<	66 Dy	<
				67+Ho	<
				68+Er	<
				69+Tm	<
				70+Yb	0.57 0.26
				71 Lu	<

```
KnownConc= 0                  REST= 0              D/S= 0
Sum Conc's before normalisation to 100% :  101.9 %
```

Table 32

```
Quantum Catalytics, LLC        ANALYSIS REPORT              by Uniquant
---------------------------------------------------------------------------
OLD.615 of  7-Mar-02                                    Today  7-Mar-02
Spectrometers configuration:  ARL 8410 Rh 60kV LiF220 LiF420 Ge111 TlAP
Sample ident   = 14-00-01 AX
Further info   = 14-00-01 AXIAL POLISHED VAC 3/7/02
Kappa list     = 15-Nov-94    Channel list =   8-Jan-02
Calculated as  : Elements     Spectral impurity data : CAL.909 Teflon (7/94)
X-ray path     = Vacuum       Film type    = No supporting film
Case number    = 0   Known Area, %Rest, Diluent/Sample and Mass/Area
Eff.Diam.      = 25.00 mm      Eff.Area     = 490.6 mm2
KnownConc      = 0   %
Rest           = 0   %
Dil/Sample     = 0
Viewed Mass    = 18000.00 mg
Sample Height  = 5   mm
            <     means that the concentration is <  20 ppm
            <2e   means wt% < 2 StdErr. The + in Z+El means involved in Sum=100%
     Z      wt%     StdErr     Z        wt%     StdErr     Z         wt%     StdErr
  ===========================  =========================  ===========================
  SumBe..F  0       0.043    29+Cu    98.88    0.05     51 Sb       <
  11+Na     0.063   0.012    30+Zn    <                 52 Te       <
  12 Mg     <                31+Ga    0.008    0.003    53 I        <
  13+Al     0.33    0.02     32 Ge    <                 55 Cs       <
  14+Si     0.66    0.03     33 As    <                 56 Ba       <

  15+P      <                34 Se    <                 SumLa..Lu   0.021   0.073
  16+S      0.0042  0.0004   35 Br    <                 72+Hf       <
  16 So                      37 Rb    <                 73+Ta       <
  17+Cl     0.026   0.002    38 Sr    <                 74 W        <
  18+Ar     0.013   0.001    39 Y     <                 75 Re       <

  19 K      <                40 Zr    <                 76 Os       <
  20 Ca     <                41 Nb    <                 77 Ir       <2e     0.008
  21+Sc     <                42 Mo    <2e      0.002    78 Pt       <
  22 Ti     <                44 Ru    <                 79 Au       <
  23 V      <                45 Rh    <2e      0.002    80 Hg       <2e     0.006

  24 Cr     <                46 Pd    <                 81 Tl       <
  25 Mn     <                47 Ag    <2e      0.002    82 Pb       <
  26 Fe     <                48 Cd    <2e      0.002    83 Bi       <
  27 Co     <                49 In    <                 90 Th       <
  28 Ni     <                50 Sn    <                 92 U        <

  ==== Light Elements =====  ==== Noble Elements =====  ===== Lanthanides =======
  4 Be                       44 Ru    <                 57+La       0.012   0.005
  5 B                        45 Rh    <2e      0.002    58 Ce       <
  6 C                        46 Pd    <                 59 Pr       <
  7 N                        47 Ag    <2e      0.002    60 Nd       <
  8 O                        75 Re    <                 62 Sm       <
  9 F       <                76 Os    <                 63 Eu       <
                             77 Ir    <2e      0.008    64 Gd       <
                             78 Pt    <                 65 Tb       <2e     0.002
                             79 Au    <                 66 Dy       <
                                                        67 Ho       <
                                                        68 Er       <2e     0.004
                                                        69 Tm       <2e     0.004
                                                        70 Yb       <
                                                        71+Lu       <

  KnownConc= 0                     REST= 0                   D/S= 0
  Sum Conc's before normalisation to 100% :   100.0 %
```

Table 33

```
Quantum Catalytics, LLC          ANALYSIS REPORT                    by Uniquant
-------------------------------------------------------------------------------
OLD.620 of  8-Jan-02                                        Today  7-Mar-02
Spectrometers configuration:  ARL 8410 Rh 60kV LiF220 LiF420 Ge111 TlAP
Sample ident   =  14-00-01 RAD
Further info   =  14-00-01 RADIAL POLISHED VAC 3/7/02
Kappa list     =  15-Nov-94    Channel list =    8-Jan-02
Calculated as  :  Elements     Spectral impurity data : CAL.909 Teflon (7/94)
X-ray path     =  Vacuum       Film type     = No supporting film
Case number    =  0   Known Area, %Rest, Diluent/Sample and Mass/Area
Eff.Diam.      = 25.00 mm       Eff.Area      = 490.6 mm2
KnownConc      =  0    %
Rest           =  0    %
Dil/Sample     =  0
Viewed Mass    = 18000.00 mg
Sample Height  =  5   mm
             <     means that the concentration is <  20 ppm
             <2e  means wt% < 2 StdErr. The + in Z+El means involved in Sum=100%
```

Z	wt%	StdErr	Z	wt%	StdErr	Z	wt%	StdErr
SumBe..F	0.11	0.04	29+Cu	98.84	0.05	51 Sb	<	
11+Na	0.11	0.01	30+Zn	<		52 Te	<	
12 Mg	<		31 Ga	<2e	0.003	53 I	<	
13+Al	0.28	0.02	32 Ge	<		55 Cs	<2e	0.003
14+Si	0.58	0.03	33 As	<		56 Ba	0.006	0.003
			34 Se	<		SumLa..Lu	0.011	0.072
15+P	<		35 Br	<		72+Hf	<	
16+S	0.0065	0.0006	37 Rb	<		73+Ta	<	
16 So			38 Sr	<		74 W	<	
17+Cl	0.029	0.002	39 Y	<		75 Re	<2e	0.007
18+Ar	0.015	0.001						
19+K	0.0021	0.0007	40 Zr	<		76 Os	<2e	0.007
20+Ca	0.0041	0.0008	41 Nb	<		77+Ir	0.018	0.008
21 Sc	<		42+Mo	<2e	0.002	78 Pt	<2e	0.008
22 Ti	<2e	0.001	44 Ru	<2e	0.002	79 Au	<	
23 V	<		45 Rh	<		80 Hg	<	
24 Cr	<		46 Pd	<		81 Tl	<	
25 Mn	<		47 Ag	<2e	0.002	82 Pb	<	
26 Fe	<		48 Cd	<2e	0.002	83 Bi	<	
27+Co	<		49 In	<		90 Th	<2e	0.003
28 Ni	<		50 Sn	<		92 U	<	

==== Light Elements =====			==== Noble Elements =====			===== Lanthanides =======		
4 Be			44 Ru	<2e	0.002	57 La	<2e	0.005
5 B			45 Rh	<		58 Ce	<	
6 C			46 Pd	<		59 Pr	<	
7 N			47 Ag	<2e	0.002	60 Nd	<	
8 O			75 Re	<2e	0.007	62 Sm	<	
9+F	0.11	0.04	76 Os	<2e	0.007	63 Eu	<2e	0.002
			77+Ir	0.018	0.008	64 Gd	<	
			78 Pt	<2e	0.008	65 Tb	<2e	0.002
			79 Au	<		66 Dy	<	
						67 Ho	<	
						68 Er	<	
						69 Tm	<2e	0.004
						70 Yb	<	
						71+Lu	<	

```
KnownConc= 0                      REST= 0                 D/S= 0
Sum Conc's before normalisation to 100% :   99.8 %
```

Table 34

```
Quantum Catalytics, LLC          ANALYSIS REPORT                  by Uniquant
-----------------------------------------------------------------------------
OLD.613 of 7-Mar-02                                     Today  7-Mar-02
Spectrometers configuration:  ARL 8410 Rh 60kV LiF220 LiF420 Ge111 TlAP
Sample ident   =  14-00-03 AX
Further info   =  14-00-03 AXIAL POLISHED VAC 3/7/02
Kappa list     =  15-Nov-94     Channel list =   8-Jan-02
Calculated as :   Elements      Spectral impurity data : CAL.909 Teflon (7/94)
X-ray path     =  Vacuum        Film type    = No supporting film
Case number    =  0   Known Area, %Rest, Diluent/Sample and Mass/Area
Eff.Diam.      = 25.00 mm        Eff.Area     = 490.6 mm2
KnownConc      =  0      %
Rest           =  0      %
Dil/Sample     =  0
Viewed Mass    = 18000.00 mg
Sample Height =   5   mm
              <     means that the concentration is <  20 ppm
              <2e  means wt% < 2 StdErr. The + in Z+El means involved in Sum=100%
```

Z	wt%	StdErr	Z	wt%	StdErr	Z	wt%	StdErr
SumBe..F	0	0.043	29+Cu	99.69	0.03	51 Sb	<	
11+Na	0.039	0.012	30+Zn	<		52 Te	<	
12 Mg	<		31 Ga	<2e	0.003	53 I	<	
13+Al	0.103	0.008	32 Ge	<2e	0.002	55 Cs	<	
14+Si	0.036	0.006	33 As	<		56 Ba	<	
15+P	<		34 Se	<		SumLa..Lu	0.033	0.071
16+S	0.0056	0.0005	35 Br	<		72+Hf	<	
16 So			37 Rb	<		73+Ta	<	
17+Cl	0.037	0.003	38 Sr	<		74 W	<	
18+Ar	0.013	0.001	39 Y	<		75 Re	<	
19+K	<		40 Zr	<		76 Os	<	
20+Ca	0.0048	0.0008	41 Nb	<		77+Ir	0.031	0.008
21 Sc	<		42+Mo	0.005	0.002	78 Pt	<	
22+Ti	0.003	0.001	44 Ru	<		79 Au	<2e	0.007
23 V	<		45 Rh	<		80 Hg	<	
24 Cr	<		46 Pd	<		81 Tl	<	
25 Mn	<		47 Ag	<		82 Pb	<	
26 Fe	<		48 Cd	<2e	0.002	83 Bi	<	
27+Co	<		49 In	<		90 Th	<	
28 Ni	<		50 Sn	<		92 U	<	

==== Light Elements =====		==== Noble Elements =====		===== Lanthanides ======		
4 Be		44 Ru	<	57+La	0.015	0.005
5 B		45 Rh	<	58 Ce	<	
6 C		46 Pd	<	59 Pr	<	
7 N		47 Ag	<	60 Nd	<	
8 O		75 Re	<	62 Sm	<	
9 F	<	76 Os	<	63 Eu	<2e	0.002
		77+Ir	0.031	0.008	64 Gd	<
		78 Pt	<	65 Tb	<	
		79 Au	<2e	0.007	66 Dy	<
				67 Ho	<	
				68+Er	0.015	0.004
				69 Tm	<	
				70 Yb	<	
				71+Lu	<	

```
KnownConc= 0                    REST= 0              D/S= 0
Sum Conc's before normalisation to 100% :    99.4 %
```

Table 35

```
Quantum Catalytics, LLC        ANALYSIS REPORT              by Uniquant
-----------------------------------------------------------------------
OLD.618 of  8-Jan-02                                    Today  7-Mar-02
Spectrometers configuration: ARL 8410 Rh 60kV LiF220 LiF420 Ge111 TlAP
Sample ident   =   14-00-03 RAD
Further info   =   14-00-03 RADIAL POLISHED VAC 3/7/02
Kappa list     =   15-Nov-94    Channel list =  8-Jan-02
Calculated as :    Elements     Spectral impurity data : CAL.909 Teflon (7/94)
X-ray path     =   Vacuum       Film type    = No supporting film
Case number    =   0   Known Area, %Rest, Diluent/Sample and Mass/Area
Eff.Diam.      = 25.00 mm        Eff.Area     = 490.6 mm2
KnownConc      =   0     %
Rest           =   0     %
Dil/Sample     =   0
Viewed Mass    = 18000.00 mg
Sample Height  =   5   mm
             <    means that the concentration is <   20 ppm
             <2e  means wt% < 2 StdErr. The + in Z+El means involved in Sum=100%
   Z      wt%     StdErr     Z           wt%    StdErr     Z          wt%    StdErr
  ========================   ========================   ========================
SumBe..F    0      0.042   29+Cu    98.93   0.05    51 Sb      <
11+Na    0.23      0.02    30+Zn    <               52 Te      <
12 Mg    <                 31+Ga    0.008   0.003   53 I       <
13+Al    0.37      0.02    32 Ge    <2e     0.003   55 Cs      <
14+Si    0.29      0.02    33 As    <               56 Ba      <

15+P     0.004     0.001   34 Se    <               SumLa..Lu  0.008   0.072
16+S     0.021     0.002   35 Br    <               72+Hf      <
16 So                      37 Rb    <               73+Ta      <
17+Cl    0.068     0.006   38 Sr    <2e     0.001   74 W       <
18+Ar    0.018     0.002   39 Y     <               75 Re      <

19+K     0.013     0.001   40 Zr    <               76 Os      <
20+Ca    0.017     0.002   41 Nb    <               77+Ir      0.023   0.008
21+Sc    0.0036    0.0009  42 Mo    <               78 Pt      <
22 Ti    <                 44 Ru    <               79 Au      <
23 V     <                 45 Rh    <2e     0.002   80 Hg      <2e     0.007

24 Cr    <                 46 Pd    <               81 Tl      <2e     0.005
25 Mn    <                 47+Ag    0.004   0.002   82 Pb      <2e     0.003
26 Fe    <                 48 Cd    <2e     0.002   83 Bi      <
27 Co    <                 49 In    <               90 Th      <
28 Ni    <                 50 Sn    <               92 U       <

==== Light Elements =====   ==== Noble Elements =====   ===== Lanthanides ======
 4 Be                       44 Ru    <               57 La      <
 5 B                        45 Rh    <2e     0.002   58 Ce      <
 6 C                        46 Pd    <               59 Pr      <
 7 N                        47+Ag    0.004   0.002   60 Nd      <
 8 O                        75 Re    <               62 Sm      <
 9 F     <                  76 Os    <               63 Eu      <
                            77+Ir    0.023   0.008   64 Gd      <
                            78 Pt    <               65 Tb      <2e     0.002
                            79 Au    <               66 Dy      <
                                                     67 Ho      <
                                                     68 Er      <2e     0.004
                                                     69 Tm      <
                                                     70 Yb      <
                                                     71+Lu      <

KnownConc= 0                    REST= 0                    D/S= 0
Sum Conc's before normalisation to 100% :    90.0 %
```

Table 36

```
Quantum Catalytics, LLC          ANALYSIS REPORT                    by Uniquant
--------------------------------------------------------------------------------
OLD.612 of  7-Mar-02                                      Today  7-Mar-02
Spectrometers configuration:  ARL 8410 Rh 60kV LiF220 LiF420 Ge111 TlAP
Sample ident   =  14-00-04 AX
Further info   =  14-00-04 AXIAL POLISHED VAC 3/7/02
Kappa list     =  15-Nov-94    Channel list  =   8-Jan-02
Calculated as :   Elements     Spectral impurity data : CAL.909 Teflon (7/94)
X-ray path     =  Vacuum       Film type     = No supporting film
Case number    =  0   Known Area, %Rest, Diluent/Sample and Mass/Area
Eff.Diam.      = 25.00 mm       Eff.Area      = 490.6 mm2
KnownConc      =  0     %
Rest           =  0     %
Dil/Sample     =  0
Viewed Mass    = 18000.00 mg
Sample Height  =  5   mm
             <     means that the concentration is <  20 ppm
             <2e   means wt% < 2 StdErr. The + in Z+El means involved in Sum=100%
```

Z	wt%	StdErr	Z	wt%	StdErr	Z	wt%	StdErr
SumBe..F	0	0.042	29+Cu	99.29	0.05	51 Sb	<	
11+Na	0.070	0.011	30+Zn	<		52 Te	<	
12 Mg	<		31 Ga	<2e	0.003	53 I	<	
13+Al	0.27	0.02	32 Ge	<		55 Cs	<2e	0.003
14+Si	0.29	0.02	33 As	<		56 Ba	<2e	0.003
15+P	<		34 Se	<		SumLa..Lu	0.023	0.072
16 S			35 Br	<		72+Hf	<2e	0.022
16+So	0.0074	0.0007	37 Rb	<		73+Ta	<	
17+Cl	0.021	0.002	38 Sr	<		74 W	<	
18+Ar	0.012	0.001	39 Y	<		75 Re	<	
19+K	0.0023	0.0007	40 Zr	<		76 Os	<	
20 Ca	<		41 Nb	<		77+Ir	0.026	0.008
21 Sc	<		42 Mo	<		78 Pt	<2e	0.007
22+Ti	<2e	0.001	44 Ru	<		79 Au	<	
23 V	<		45 Rh	<		80 Hg	<2e	0.006
24 Cr	<		46 Pd	<		81 Tl	<	
25 Mn	<		47 Ag	<		82 Pb	<	
26 Fe	<		48 Cd	<		83 Bi	<	
27 Co	<		49 In	<		90 Th	<	
28 Ni	<		50 Sn	<		92 U	<	

```
==== Light Elements =====    ==== Noble Elements =====    ===== Lanthanides =======
   4 Be                        44 Ru   <                   57+La    0.013   0.005
   5 B                         45 Rh   <                   58 Ce    <
   6 C                         46 Pd   <                   59 Pr    <
   7 N                         47 Ag   <                   60 Nd    <
   8 O                         75 Re   <                   62 Sm    <
   9 F       <                 76 Os   <                   63 Eu    <
                               77+Ir   0.026   0.008       64 Gd    <
                               78 Pt   <2e     0.007       65 Tb    <2e     0.002
                               79 Au   <                   66 Dy    <
                                                           67 Ho    <
                                                           68 Er    <2e     0.004
                                                           69 Tm    <2e     0.004
                                                           70 Yb    <
                                                           71+Lu    <
```

```
KnownConc= 0                    REST= 0                    D/S= 0
Sum Conc's before normalisation to 100% :    99.5 %
```

Table 37

```
Quantum Catalytics, LLC          ANALYSIS REPORT                by Uniquant
---------------------------------------------------------------------------
OLD.619 of  8-Jan-02                                  Today  7-Mar-02
Spectrometers configuration:  ARL 8410 Rh 60kV LiF220 LiF420 Ge111 TlAP
Sample ident  =  14-00-04 RAD
Further info  =  14-00-04 RADIAL POLISHED VAC 3/7/02
Kappa list    =  15-Nov-94    Channel list =   8-Jan-02
Calculated as :  Elements     Spectral impurity data : CAL.909 Teflon (7/94)
X-ray path    =  Vacuum       Film type     = No supporting film
Case number   =  0    Known Area, %Rest, Diluent/Sample and Mass/Area
Eff.Diam.     =  25.00 mm       Eff.Area      = 490.6 mm2
KnownConc     =  0     %
Rest          =  0     %
Dil/Sample    =  0
Viewed Mass   =  18000.00 mg
Sample Height =  5   mm
              <    means that the concentration is <  20 ppm
              <2e  means wt% < 2 StdErr. The + in Z+El means involved in Sum=100%
   Z      wt%      StdErr     Z       wt%     StdErr     Z          wt%      StdErr
=========================   =========================   ===========================
SumBe..F  0        0.044    29+Cu   98.59    0.06     51 Sb       <
11+Na     0.062    0.012    30+Zn   <                 52 Te       <
12 Mg     <                 31 Ga   <2e      0.003    53 I        <
13+Al     0.63     0.03     32 Ge   <                 55 Cs       <
14+Si     0.60     0.03     33 As   <                 56 Ba       <

15+P      <                 34 Se   <                 SumLa..Lu   0.021    0.073
16+S      0.012    0.001    35 Br   <                 72+Hf       <2e      0.022
16 So                       37 Rb   <                 73+Ta       <
17+Cl     0.020    0.002    38 Sr   <                 74 W        <
18+Ar     0.016    0.002    39 Y    <                 75 Re       <2e      0.009

19+K      <                 40+Zr   0.005    0.001    76 Os       <
20+Ca     0.0109   0.0010   41 Nb   <                 77+Ir       0.035    0.009
21 Sc     <                 42 Mo   <                 78 Pt       <
22+Ti     0.0037   0.0010   44 Ru   <2e      0.002    79 Au       <
23 V      <                 45 Rh   <                 80 Hg       <2e      0.006

24+Cr     0.0020   0.0006   46 Pd   <                 81 Tl       <
25 Mn     <                 47 Ag   <                 82 Pb       <
26 Fe     <                 48 Cd   <2e      0.002    83 Bi       <
27 Co     <                 49 In   <                 90 Th       <2e      0.003
28 Ni     <                 50 Sn   <                 92 U        <

==== Light Elements =====   ==== Noble Elements =====   ===== Lanthanides =======
 4 Be                       44 Ru   <2e      0.002    57+La       0.010    0.004
 5 B                        45 Rh   <                 58 Ce       <
 6 C                        46 Pd   <                 59 Pr       0.005    0.002
 7 N                        47 Ag   <                 60 Nd       <
 8 O                        75 Re   <2e      0.009    62 Sm       <
 9 F      <                 76 Os   <                 63 Eu       <
                            77+Ir   0.035    0.009    64 Gd       <2e      0.002
                            78 Pt   <                 65 Tb       <2e      0.002
                            79 Au   <                 66 Dy       <
                                                      67 Ho       <
                                                      68 Er       <
                                                      69 Tm       <
                                                      70 Yb       <
                                                      71+Lu       <

KnownConc= 0                        REST= 0                    D/S= 0
Sum Conc's before normalisation to 100% :    88.1 %
```

Table 38

```
Quantum Catalytics, LLC          ANALYSIS REPORT              by Uniquant
------------------------------------------------------------------------
OLD.614 of 7-Mar-02                               Today  7-Mar-02
Spectrometers configuration:  ARL 8410 Rh 60kV LiF220 LiF420 Ge111 T1AP
Sample ident   =  15-00-01 AX
Further info   =  15-00-01 AXIAL POLISHED VAC 3/7/02
Kappa list     =  15-Nov-94    Channel list =   8-Jan-02
Calculated as :   Elements     Spectral impurity data : CAL.909 Teflon (7/94)
X-ray path     =  Vacuum       Film type    = No supporting film
Case number    =  0   Known Area, %Rest, Diluent/Sample and Mass/Area
Eff.Diam.      = 25.00 mm       Eff.Area     = 490.6 mm2
KnownConc      =  0     %
Rest           =  0     %
Dil/Sample     =  0
Viewed Mass    = 18000.00 mg
Sample Height  =  5    mm
            <     means that the concentration is <  20 ppm
            <2e   means wt% < 2 StdErr. The + in Z+El means involved in Sum=100%
   Z      wt%      StdErr     Z       wt%     StdErr     Z         wt%     StdErr
========================    ========================    ==========================
SumBe..F  0        0.043    29+Cu  99.21     0.04     51 Sb      <
11+Na     0.048    0.012    30+Zn     <               52 Te      <
12 Mg     <                 31+Ga     0.008   0.003    53 I       <
13+Al     0.29     0.02     32 Ge     <2e     0.002    55 Cs      <2e     0.003
14+Si     0.37     0.02     33 As     <               56 Ba      <

15+P      <                 34 Se     <                SumLa..Lu  0.011   0.072
16+S      0.0043   0.0004   35 Br     <               72+Hf      <2e     0.022
16 So                       37 Rb     <               73+Ta      <
17+Cl     0.021    0.002    38 Sr     <               74 W       <
18+Ar     0.014    0.001    39 Y      <               75 Re      <2e     0.008

19+K      <                 40 Zr     <               76 Os      <2e     0.007
20 Ca     <                 41 Nb     <               77+Ir      0.036   0.008
21+Sc     0.0021   0.0009   42 Mo     <               78 Pt      <
22 Ti     <                 44 Ru     <               79 Au      <2e     0.007
23 V      <                 45 Rh     <               80 Hg      <

24 Cr     <                 46 Pd     <               81 Tl      <
25 Mn     <                 47 Ag     <               82 Pb      <
26 Fe     <                 48 Cd     <               83 Bi      <
27 Co     <                 49 In     <               90 Th      <
28 Ni     <                 50 Sn     <               92 U       <

==== Light Elements =====   ==== Noble Elements =====  ===== Lanthanides =======
 4 Be                       44 Ru     <               57 La      <2e     0.005
 5 B                        45 Rh     <               58 Ce      <
 6 C                        46 Pd     <               59 Pr      <
 7 N                        47 Ag     <               60 Nd      <
 8 O                        75 Re     <2e     0.008   62 Sm      <
 9 F      <                 76 Os     <2e     0.007   63 Eu      <
                            77+Ir     0.036   0.008   64 Gd      <
                            78 Pt     <               65 Tb      <
                            79 Au     <2e     0.007   66 Dy      <
                                                      67 Ho      <
                                                      68 Er      <2e     0.004
                                                      69 Tm      <
                                                      70 Yb      <
                                                      71+Lu      <


KnownConc= 0                     REST= 0                    D/S= 0
Sum Conc's before normalisation to 100% :   99.7 %
```

Table 39

```
Quantum Catalytics, LLC            ANALYSIS REPORT                    by Uniquant
--------------------------------------------------------------------------------
OLD.617 of  8-Jan-02                                          Today  7-Mar-02
Spectrometers configuration:  ARL 8410 Rh 60kV LiF220 LiF420 Ge111 TlAP
Sample ident   =  15-00-01 RAD
Further info   =  15-00-01 RADIAL POLISHED VAC 3/7/02
Kappa list     =  15-Nov-94    Channel list =    8-Jan-02
Calculated as  :  Elements     Spectral impurity data : CAL.909 Teflon (7/94)
X-ray path     =  Vacuum       Film type    = No supporting film
Case number    =  0   Known Area, %Rest, Diluent/Sample and Mass/Area
Eff.Diam.      = 25.00 mm       Eff.Area     = 490.6 mm2
KnownConc      =  0     %
Rest           =  0     %
Dil/Sample     =  0
Viewed Mass    = 18000.00 mg
Sample Height  =  5   mm
```

```
             <     means that the concentration is <  20 ppm
             <2e   means wt% < 2 StdErr. The + in Z+El means involved in Sum=100%
   Z       wt%     StdErr    Z          wt%     StdErr    Z           wt%     StdErr
========================  ========================  ========================
SumBe..F  0       0.041    29+Cu    99.39    0.05     51 Sb       <
11+Na     0.093   0.012    30+Zn    <                 52 Te       <
12 Mg     <                31+Ga    0.007    0.003    53 I        <
13+Al     0.33    0.02     32 Ge    <2e      0.002    55 Cs       <
14+Si     0.068   0.006    33 As    <                 56 Ba       <

15+P      <                34 Se    <                 SumLa..Lu   0.017   0.073
16+S      0.0083  0.0007   35 Br    <                 72+Hf       <
16 So                      37 Rb    <                 73+Ta       <
17+Cl     0.034   0.003    38 Sr    <                 74 W        <
18+Ar     0.017   0.002    39 Y     <                 75 Re       <

19+K      0.0053  0.0008   40 Zr    <                 76 Os       <
20+Ca     0.0036  0.0008   41 Nb    <                 77+Ir       0.031   0.008
21 Sc     <                42 Mo    <2e      0.002    78 Pt       <
22 Ti     <                44 Ru    <2e      0.002    79 Au       <2e     0.007
23 V      <                45 Rh    <                 80 Hg       <2e     0.006

24 Cr     <                46 Pd    <                 81+Tl       0.014   0.005
25 Mn     <                47 Ag    <                 82 Pb       <
26 Fe     <                48 Cd    <                 83 Bi       <2e     0.005
27 Co     <                49 In    <                 90 Th       <
28 Ni     <                50 Sn    <                 92 U        <

==== Light Elements =====  ==== Noble Elements =====  ===== Lanthanides =======
 4 Be                      44 Ru    <2e      0.002    57 La       <2e     0.005
 5 B                       45 Rh    <                 58 Ce       <
 6 C                       46 Pd    <                 59 Pr       <
 7 N                       47 Ag    <                 60 Nd       <
 8 O                       75 Re    <                 62 Sm       <
 9 F      <                76 Os    <                 63 Eu       <2e     0.002
                           77+Ir    0.031    0.008    64 Gd       <2e     0.002
                           78 Pt    <                 65 Tb       <
                           79 Au    <2e      0.007    66 Dy       <
                                                      67 Ho       <
                                                      68 Er       <2e     0.004
                                                      69 Tm       <2e     0.004
                                                      70 Yb       <
                                                      71+Lu       <

KnownConc= 0                    REST= 0                    D/S= 0
Sum Conc's before normalisation to 100% :    94.6 %
```

Table 40

```
Quantum Catalytics, LLC          ANALYSIS REPORT              by Uniquant
------------------------------------------------------------------------------
OLD.340 of 11-Jun-01                                       Today 11-Jun-01
Spectrometers configuration: ARL 8410 Rh 60kV LiF220 LiF420 Ge111 TlAP
Sample ident   =  15-01-03    AXIAL
Further info   =  15-01-03 AXIAL Fe,Cr,Mn,V 6/11
Kappa list     =  15-Nov-94    Channel list =   4-Jun-01
Calculated as :  Elements     Spectral impurity data : CAL.909 Teflon (7/94)
X-ray path     =  Vacuum       Film type     = No supporting film
Case number    =  0   Known Area, %Rest, Diluent/Sample and Mass/Area
Eff.Diam.      = 25.00 mm      Eff.Area      = 490.6 mm2
KnownConc      =  0     %
Rest           =  0     %
Dil/Sample     =  0
Viewed Mass    = 18000.00 mg
Sample Height  =  5  mm
             <    means that the concentration is <  20 ppm
            <2e   means wt% < 2 StdErr. The + in Z+El means involved in Sum=100%
   z      wt%      StdErr   z        wt%     StdErr    z        wt%      StdErr
========================= ========================= =========================
SumBe..F     0     0.039   29+Cu    0.028   0.002   51 Sb      <
11+Na     0.032   0.009    30+Zn    0.005   0.002   52 Te      <
12 Mg     <                31 Ga    <2e     0.001   53 I       <2e      0.002
13+Al     2.28    0.07     32 Ge    <               55 Cs      <2e      0.002
14 Si     <                33 As    <               56+Ba      <2e      0.003

15+P      0.0038  0.0003   34 Se    <               SumLa..Lu  0        0.21
16 S                       35 Br    <               72 Hf      <2e      0.004
16+So     0.0092  0.0008   37 Rb    <               73 Ta      <
17+Cl     0.017   0.002    38 Sr    <               74 W       <2e      0.004
18+Ar     0.009   0.001    39 Y     <               75 Re      <

19+K      <                40+Zr    0.045   0.004   76 Os      <
20+Ca     0.0060  0.0006   41 Nb    <               77 Ir      <
21 Sc     <                42 Mo    <               78 Pt      <
22 Ti     <                44 Ru    <               79 Au      <
23+V      2.38    0.07     45 Rh    <               80 Hg      <

24+Cr     2.71    0.08     46 Pd    <               81 Tl      <
25+Mn     2.05    0.07     47 Ag    <               82 Pb      <
26+Fe     90.4    0.1      48 Cd    <               83 Bi      <
27 Co     <                49 In    <               90+Th      0.005    0.002
28+Ni     0.012   0.003    50 Sn    <               92 U       <

==== Light Elements =====  ==== Noble Elements =====  ===== Lanthanides ======
 4 Be                      44 Ru    <               57 La      <
 5 B                       45 Rh    <               58 Ce      <
 6 C                       46 Pd    <               59+Pr      <
 7 N                       47 Ag    <               60 Nd      <
 8 O                       75 Re    <               62+Sm      <
 9 F      <                76 Os    <               63+Eu      <
                           77 Ir    <               64 Gd      <
                           78 Pt    <               65+Tb      <
                           79 Au    <               66 Dy      <
                                                     67 Ho      <
                                                     68+Er      <
                                                     69+Tm      <
                                                     70 Yb      <
                                                     71 Lu      <

KnownConc= 0                       REST= 0                 D/S= 0
Sum Conc's before normalisation to 100% :  100.1 %
```

Table 41

```
Quantum Catalytics, LLC        ANALYSIS REPORT              by Uniquant
------------------------------------------------------------------------------
OLD.341 of  4-Jun-01                                   Today 11-Jun-01
Spectrometers configuration: ARL 8410 Rh 60kV LiF220 LiF420 Ge111 TlAP
Sample ident  = 15-01-03    RADIAL
Further info  = 15-01-03 RADIAL Fe,Cr,Mn,V 6/11
Kappa list    = 15-Nov-94   Channel list =   4-Jun-01
Calculated as :  Elements   Spectral impurity data : CAL.909 Teflon (7/94)
X-ray path    = Vacuum      Film type    = No supporting film
Case number   = 0   Known Area, %Rest, Diluent/Sample and Mass/Area
Eff.Diam.     = 25.00 mm      Eff.Area     = 490.6 mm2
KnownConc     = 0    %
Rest          = 0    %
Dil/Sample    = 0
Viewed Mass   = 18000.00 mg
Sample Height =  5   mm
              <    means that the concentration is <  20 ppm
              <2e  means wt% < 2 StdErr. The + in Z+El means involved in Sum=100%
   Z       wt%    StdErr    Z        wt%    StdErr    Z          wt%    StdErr
========================= ========================= ============================
SumBe..F   0      0.036   29+Cu     0.16    0.01    51 Sb       <
11+Na      0.083  0.009   30+Zn     0.032   0.003   52 Te       <
12+Mg      0.013  0.003   31 Ga     <2e     0.001   53 I        <
13+Al      4.5    0.1     32 Ge     <               55 Cs       <
14+Si      0.15   0.01    33 As     <               56 Ba       <

15+P       0.0097 0.0009  34 Se     <               SumLa..Lu   0      0.20
16 S                      35 Br     <               72+Hf       0.018  0.004
16+So      0.048  0.004   37 Rb     <               73 Ta       <
17+Cl      0.032  0.003   38 Sr     <               74+W        0.008  0.004
18+Ar      0.010  0.001   39 Y      <               75 Re       <

19+K       0.019  0.002   40+Zr     0.097   0.008   76 Os       <
20+Ca      0.051  0.004   41 Nb     <               77 Ir       <2e    0.003
21 Sc      <              42 Mo     <2e     0.002   78 Pt       <
22+Ti      0.0030 0.0006  44 Ru     <               79 Au       <
23+V       2.26   0.07    45 Rh     <               80 Hg       <

24+Cr      2.39   0.07    46 Pd     <               81 Tl       <
25+Mn      2.20   0.07    47 Ag     <2e     0.001   82 Pb       <
26+Fe      87.9   0.2     48 Cd     <2e     0.001   83 Bi       <
27 Co      <              49 In     <               90+Th       0.006  0.002
28 Ni      <2e    0.003   50 Sn     <               92 U        <

==== Light Elements =====  ==== Noble Elements =====  ===== Lanthanides =======
  4 Be                     44 Ru     <               57 La       <
  5 B                      45 Rh     <               58 Ce       <
  6 C                      46 Pd     <               59+Pr       <
  7 N                      47 Ag     <2e     0.001   60 Nd       <
  8 O                      75 Re     <               62+Sm       <
  9 F       <              76 Os     <               63+Eu       <
                           77 Ir     <2e     0.003   64 Gd       <
                           78 Pt     <               65+Tb       <
                           79 Au     <               66 Dy       <
                                                     67 Ho       <
                                                     68+Er       <
                                                     69+Tm       <
                                                     70 Yb       <
                                                     71 Lu       <

KnownConc= 0                   REST= 0                D/S= 0
Sum Conc's before normalisation to 100% :   99.9 %
```